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NEW ABLATIVE PLASTICS AND COMPOSITES, THEIR FORMULATION AND PROCESSING

B.G. KIMMEL AND G. SCHWARTZ
Hughes Aircraft Company

TECHNICAL REPORT AFML-TR-66-75, PART II

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AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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FOREWORD

This report was prepared by Hughes Aircraft Company, Culver City, California, under USAF Contract No. AF 33(615)-2418. This contract was initiated under project No. 7340, "Non-Metallic and Composite Materials," Task No. 734001, "Thermally Protective Plastics and Composites." The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division. Mr. P. F. Pirrung acted as project engineer.

This report covers work from February 1966 to February 1967. Work accomplished from February 1965 to February 1966 was reported in AFML TR 66-75.

Previous work on this contract was performed under USAF Contract No. AF 33(657)-8621 and will be found in ASD TR 63-568, Part I, ML TDR 64-222 and AFML TR 65-94.

Manuscript released by the authors March 1967 for publication as an RTD Technical Report.

This technical report has been reviewed and is approved.

R. G. Spain

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ABSTRACT

Precise processing techniques were used in preparing new ablative plastics composites. This research involved the use of novel heat-resistant resins such as:

- branched, cross-linked polyphenylenes
- para-polyphenylenes
- polyphenylene oxide
- amide-blocked polybenzimidazole
- phenyl aldehyde
- phosphonitrilic-modified phenolic
- chrome based metal organic phenolic
- tungsten based metal organic phenolic
- polyimide
- diphenyl oxide

Novel reinforcements included:

- boron fibers
- boron nitride fibers
- pyrolytic graphite fibers

Resin impregnation techniques used in preparing research specimens included spatula or brush coating, dip coating, Buchner funnel impregnation and dry powder layup.

Research specimens of controlled composition were prepared and submitted to the Air Force Materials Laboratory for hyperthermal evaluation, as follows:

- pellet specimens, 3/4-inch diameter by 1/2-inch long
- rocket nozzle assemblies
- hot gas flow specimens
- laminates, 6 x 6 x 1/8 inch
- laminated squares, 2 x 2 x 1/2 inch

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INTRODUCTION

New polymeric materials and reinforcements have been developed in government and industry research programs. Many of these materials offer considerable promise for use in high-performance ablative plastics.

The program objectives are to select promising ablative materials for further study and develop suitable fabrication procedures for preparing small ablative composites containing these new materials.

The work done during this 12 month period of the program consisted mainly of the continued use of precise processing techniques in fabricating research specimens of closely controlled composition. Specimens were produced consistently with a resin content within a ±2 percent range. In all experiments, all pertinent processing information and data were recorded to allow later duplication of any test specimens required for further tests. These processing data can be used in scaling up the processes if required.

Specimens prepared under this contract have been forwarded to the Nonmetallic Materials Division, Air Force Materials Laboratory, for subsequent hyperthermal evaluation.

Newly developed resins and reinforcements, which are becoming available in research quantities, were used to fabricate ablative composite specimens. These specimens will be subsequently characterized for possible use in high speed entry and rocket exhaust environments. Materials intended for potential entry environments will be characterized with an air arc heater. This research is being performed under AF 33(615)3923 with the Avco Corporation, SSD. Rocket nozzle specimens will be characterized using a liquid propellant motor or a solid propellant motor simulator under Contract AF 33(615)1632 with the Aeronutronic Division, Philco Corporation. Hot gas flow specimens will be characterized with a solid propellant motir, under contract AF 33(615) 1631 with the Atlantic Research Corporation.

SUMMARY

Precise formulation and processing techniques were used in the preparation of ablative composites of controlled composition containing new polymeric materials and reinforcements.

Formulating, molding, and postcuring conditions were varied, as required, to produce test specimens of high quality from a wide range of resins and reinforcements. New resins investigated included:

- branched, cross-linked polyphenylenes
- para-polyphenylenes
- polyphenylene oxide
- amide-blocked polybenzimidazole
- phenyl aldehyde
- phosphonitrilic-modified phenolic
- chrome based metal organic phenolic
- tungsten based metal organic phenolic
- polyimide
- diphenyl oxide

Novel reinforcements included:

- boron fibers
- boron nitride fibers
- pyrolytic graphite fibers

In addition, a large quantity of research specimens were fabricated using standard resins such as phenolics or epoxy novolacs and standard reinforcements such as Refrasil, carbon cloth, or graphite cloth. In many cases, a standard reinforcement was combined with a new resin while a new reinforcement was combined with one of the standard resins.

During the period covered by this report, the following specimens were prepared and shipped to Air Force Materials Laboratory.

87 hot gas flow specimens

2 laminates

29 laminated squares

111 pellet specimens

25 ASD No. 4 rocket nozzle assemblies

GENERAL SPECIMEN PREPARATION PROCEDURES

GENERAL DISCUSSION

Precise formulation and processing techniques were developed and applied in the fabrication of ablative composites containing new polymeric materials and reinforcements. Five main types of test specimens were prepared and submitted under this program:

- Hot gas flow; Type D - 3.333 x 2.000 x 0.502 ±0.002 inch (each ply 3.333 x 2.000 inch)
- Laminates; 6 x 6 x 1/8 inch or 7 x 7 x 1/8 inch
- Laminated squares; 2.000 x 2.000 x 0.502 ±0.002 inch
- Pellet specimens; 0.750 inch in diameter x 0.502 ±0.002 inch long
- Rocket nozzle assemblies; ASD No. 4

A complete description of all test specimens fabricated and delivered during the period covered by this report is given in thirteen tables in the Appendix.

Tables 1, 2, 3, 4 and 5 give density, Barcol hardness, composition and a brief description of hot gas flow specimens, laminates, laminated squares, pellet specimens, and rocket nozzles.

Table 6 lists all specimens by increasing test specimen data sheet number. It also gives the specimen type, material codes, and other information such as the data requested and shipped.

Table 7 lists all test specimens according to type of reinforcement. Table 8 lists all test specimens according to type of resin.

Tables 9, 10, 11, 12 and 13 give the fabrication details for hot gas flow specimens, laminates, laminated squares, pellet specimens and nozzles.

Table 14 lists material sources for resins, reinforcements and fillers used.

The composition of the test specimens was maintained in almost all cases within the range of ±2 weight-percent of the required nominal composition. This was done by carefully controlling each step of the fabrication process from the initial coating of the reinforcement to the final postcure of the molded or laminated composite. Past experience was used in making allowance for the weight loss (change in composition) of the coated reinforcement which takes place upon drying, B-staging, curing and postcuring.

All reinforcements except glass and high silica content cloth were oven-dried two to three hours at 240°F prior to coating with resin. All subsequent calculations were based on this dry reinforcement weight. Carbon and graphite cloth have been found to lose as much as 10 weight-percent on drying.

TYPES OF IMPREGNATION

Several methods of impregnating the reinforcements were used:

- Spatula or brush coating
- Dip coating
- Soaking
- Dry powder layup

Spatula or Brush Coating

This method of impregnation was only on cloth. Fabric is cut to a size sufficient to allow the blanking or cutting out of the proper number of plies for the molding. The dry cloth is weighed and laid out on a piece of cellophane. The proper amount of resin is weighed out and thinned, if necessary, to coating consistency. The resin is poured over the fabric and uniformly distributed over the cloth with either a spatula or a one-inch wide paint brush. The impregnated material is dried on the cellophane for 15 to 20 minutes, then hung up to dry for about one hour at room temperature. After drying at 160°F for 20 to 60 minutes, the cloth is weighed and the resin content calculated. Excess resin is removed by wiping the surface with a paper tissue soaked in thinner. However, if additional resin is needed, it is added to the backside of the cloth and uniformly distributed by either spatula or brush. When the desired resin content is reached, the fabric is "B" staged to form a prepreg. The final resin content is then calculated from the final coated weight.

Dip Coating

This method of impregnation is used only on cloth. A weighed piece of dry cloth is passed through a small dipping tray repeatedly until the required amount of resin is obtained. The cloth is allowed to dry after every fourth dip when a large number of dips are required. The drying time, from 5 to 30 minutes, depends on the resin system. Dip coating is usually used in place of spatula coating under the following circumstances:

- With solutions containing small percentages of resin solids
- With viscous resin solutions with large amounts of thinner added to obtain satisfactory coating properties

- When coating carbon or graphite cloth with a solution containing a high percentage of resin solids. These types of cloth tend to powder when spatula coated with a solution with a high solids content

When the correct resin content is obtained, the cloth is "B" staged and the final resin content is calculated as indicated above.

Soaking

This method is used with yarns, filaments, or fibers which wet readily. The dry material is placed in a beaker and thinned resin solution containing a weighed amount of resin solids is added. The reinforcement is allowed to soak for 60 minutes in air before the excess solvent is removed by evaporation under vacuum. After drying in an oven at 160°F for 60 minutes, the resin content of impregnated reinforcement is calculated from the increase in weight.

The resin content of the prepreg is increased or decreased when required, by pouring additional resin or solvent over the material and filtering off the excess. The material is "B" staged after obtaining the proper resin content.

A slight modification of this method is referred to as Buchner funnel impregnation. The dry material is placed on a piece of filter paper (Whatman's No. 4 or equivalent) in a Buchner funnel. A thinned resin solution is poured into the funnel and the material soaked for five minutes. Excess resin is then removed by vacuum filtration.

Dry Powder Layup

Cloth cannot be impregnated when the resin used is a dry insoluble powder. Specimens are prepared by sprinkling resin between plies of reinforcement. First, pieces of cloth are blanked into plies and when required are dried in an oven at 300°F for one hour. A calculated amount of resin is sprinkled between plies with each addition of resin and cloth being weighed on an analytical balance. The resin and reinforcement are weighed into a preform holder and transferred into the cold mold prior to molding.

FABRICATION PROCEDURES

Fabrication procedures for each type of specimen are given in the following sections.

HOT-GAS FLOW SPECIMENS

Type D hot gas flow specimens are machined from laminates at least 5/8-inch thick. The prepreg for molding the laminate is prepared by spatula, brush, or dip coating. After "B" staging, the material is cut into plies sufficiently large to allow the machining of the required number of specimens. The laminates are molded using the procedures described under LAMINATES and the molding conditions listed in Table 9. Specimens were rough cut prior to postcure to minimize the possibility of "blow-up." Diamond tools were used to machine the postcured pieces to final dimension.

When molding laminates, the "B" staged prepreg is cut to the proper size, randomized, stacked and wrapped in cellophane. The resulting layup is placed between 1/8-inch aluminum or 1/16-inch stainless steel cauls and loaded into the press. Laminates were cured using the molding conditions listed in Table 10. After postcure, the final resin content is determined. The laminate is then trimmed and squared by sawing with a diamond bandsaw. Finally, the density is calculated from the dimensions and weight of the specimen.

LAMINATED SQUARES

Fabrication procedures for laminated squares are the same as those for the type D hot gas flow specimens. Laminates were molded and postcured using the conditions listed in Table 11.

PELLET SPECIMENS

When sufficient material is available, pellet specimens are machined from cylindrical moldings either 2 inches or 3-1/2 inches in diameter. The parts are usually molded at least 5/8-inch thick to ensure sufficient material for machining. Individual pellets are molded when not enough material is available for molding the larger discs. Pellet specimens machined from the larger discs would be expected to vary only slightly in composition compared with specimens individually molded. Before molding a large disc, a 3/4-inch diameter pellet is made to determine the molding characteristics of the prepreg. The charge weight for the large disc is then calculated using the following formula:

$$\text{Charge weight of large disc} = \frac{\left(\text{Diameter of large disc}\right)^2 \left(\text{Desired thickness of large disc}\right) \left(\text{Weight of 3/4-inch disc}\right)}{\left(\text{Diameter of 3/4-inch disc}\right)^2 \left(\text{Thickness of 3/4-inch disc}\right)}$$

The number of plies needed to mold a laminated disc is calculated by first averaging the weights of five plies. The charge weight is then divided by the average weight per ply for the answer.

Discs were molded and postcured using the conditions listed in Table 12. The density is determined from the dimensions and weight. Pellet specimens are cut from large discs using a diamond bandsaw and all specimens are machined to final dimensions using a Carboloy cutter.

ROCKET NOZZLE SPECIMENS

The ASD No. 4 rocket nozzle assembly consists of a nozzle insert bonded into a molded phenolic housing as shown in Figure 1. All of the nozzle inserts fabricated during this report period were reinforced with plies perpendicular to the nozzle axis and were machined from blank moldings or laminates.

The method used to fabricate the nozzle insert blanks depended on the type of resin. Whenever possible, a high density, cylindrical blank was molded under high pressure in a compression mold. Some resin systems could not be cured while confined in a closed mold due to the release of excess volatiles which resulted in blistering and delamination. Materials containing such resins were laminated in an open laminating fixture which allowed the escape of excess volatiles during cure.

The nozzle housings were molded from MX2625, a heat-resistant, silica fiber and mineral-filled phenolic.

Nozzle inserts were molded and postcured using the conditions listed in Table 13. All of the internal dimensions of the inserts are machined. First, a 15/32-inch pilot hole is drilled using a tungsten carbide tool. The final configuration is then machined to the ASD No. 4 dimensions. A diamond tool on a tool post grinder is used for machining.

The density of the finished insert is determined by comparing its weight with that of an insert of known density molded from general purpose phenolic.

Nozzle inserts and nozzle housings are bonded together using Hapex 1208* containing 14-percent hardener. The bond is cured one hour at room temperature and one hour at 200°F.

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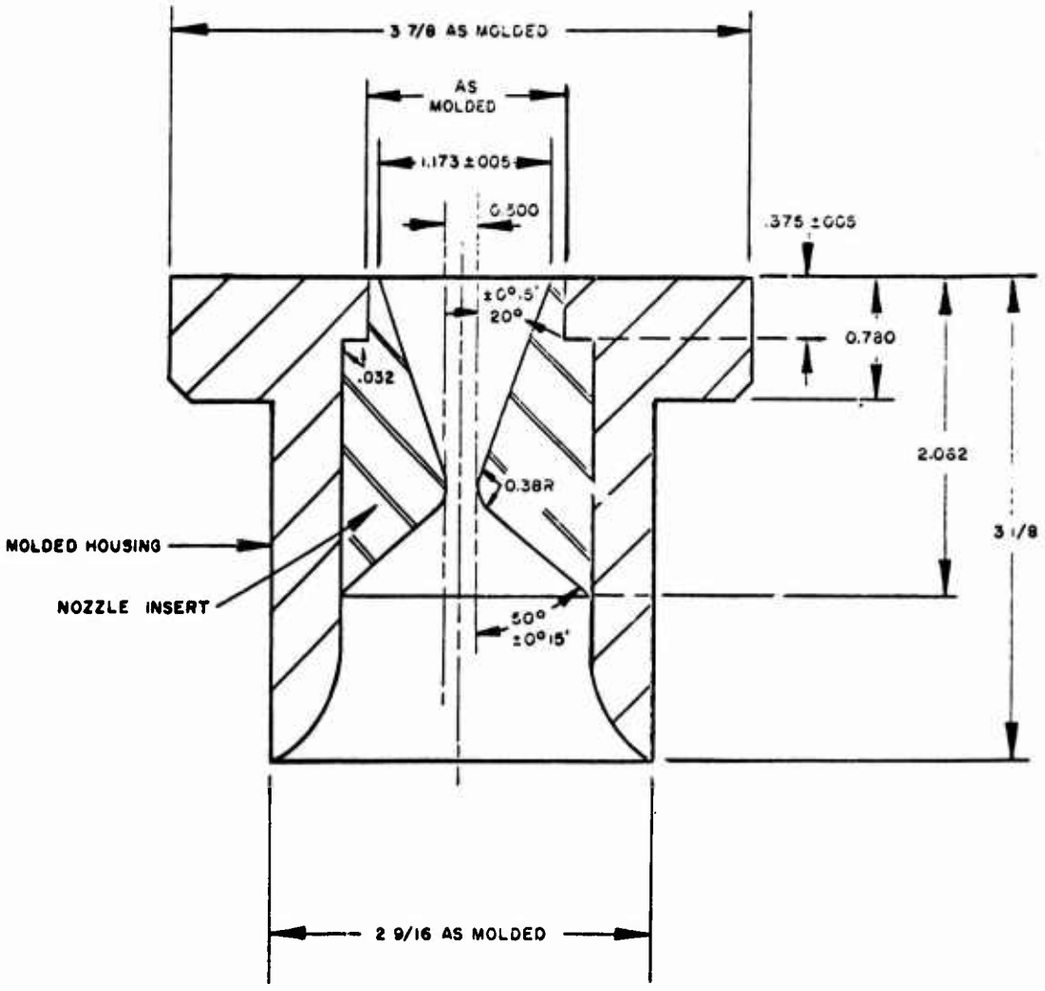
*Hastings Plastics, 1704 Colorado Blvd., Santa Monica, California.



NOZZLE INSERT OD MACHINE FOR SLIDE FIT INTO MOLDED HOUSING
INSERT BONDED INTO HOUSING WITH HAPEX 1208 (CATALYST 1213 14 PHR), CURE 1 HOUR AT ROOM TEMP 1 HOUR AT 200°F

Figure 1.

SPECIFIC SPECIMEN PROCEDURES

Certain specimens were fabricated by methods other than described in the previous section. Detailed procedures are listed below and are grouped according to specimen type.

HOT GAS FLOW SPECIMENS

- Data Sheet No. 378/394 — Polybenzimidazole (Imidite 2803 or AFR-151)-Carbon Cloth

Imidite 2803 (AFR-151) resin impregnated on carbon cloth CCA-1 was cut into 8-1/4 by 5-1/4 inch plies and molded into a 1/2-inch thick laminate. The part was cut into halves and inspected for signs of delamination. None were present. The halves were then postcured using the vendor's recommended temperature cycle. The precaution of cutting the laminate was unsuccessful since both pieces were badly delaminated when removed from the oven. The appearance of these pieces strongly indicated that they were fractured by a large evolution of gas. The heating rate in the vendor's recommended postcure appeared to be too rapid for 1/2 inch thick laminates. A second Imidite 2803 laminate, 7 by 6-1/2 bt 1/2 inches, was made and this too when sectioned proved to be of excellent quality. This laminate was then rough cut into two hot gas flow specimens, two laminated squares and six pellets, all oversize. These pieces were rough cut to minimize the possibility of "blow up" since smaller pieces have less tendency to delaminate during postcure.

Additionally, the postcure cycle recommended by the vendor was modified. This modification consisted of a much slower rate of temperature rise to allow a more gradual evolution during postcure. Unfortunately neither cutting the laminate in smaller sections nor modifying the postcure proved to be completely successful. Two of the larger pieces still "blew up." The vendor was contacted for recommendations but had none. They are aware that Imidite 2803 has the tendency to "blow up" when postcured in large sections but are unable to find a technique to prevent this. They believe the problem is inherent in the AFR-151 formulation.

One hot gas flow specimen, one laminated square and six pellets were machined from pieces not delaminated and were shipped to AFML.

- Data Sheet No. 497/488 — Polyphenylene Oxide – Graphite Cloth

Polyphenylene oxide is a thermoplastic resin which melts at approximately 600°F. It is normally considered to be an injection

molding material. Some method of coating polyphenylene oxide on cloth was necessary if a laminate was to be prepared.

The resin was received in the form of small pellets which are reported to be soluble in aromatic solvents such as toluene and xylene. A slurry of one part of PPO to three parts of xylene was prepared in a Waring blender. This slurry consisted of a dispersion of excess PPO in a PPO-xylene solution. The slurry was coated on dried graphite cloth until the cloth contained the desired amount of resin. The solvent was then removed by first air drying the prepreg followed by oven drying at 160°F and 250°F. A laminate, 2 by 2 by 1/2 inches, was successfully fabricated by wrapping stacked plies in a 6 mil aluminum foil and heating them in a 600°F press. Pressure was applied as the prepreg heated to press temperature and was just sufficient to cause a small amount of flow when the plies reached temperature. The laminate was then cooled under pressure and after being removed from the press was sawed in half for inspection. Each half in cross section appeared uniform and void free.

A large laminate, 9 by 6-3/4 by 1/2 inches, was then successfully prepared using the same procedures. The laminate was not post-cured since prolonged heating at elevated temperature does not appreciably improve the properties of thermoplastic resins. Four hot gas flow, type D specimens, four laminated squares and six pellets were machined from the laminate and shipped to AFML.

- Data Sheet No. 5[illegible]/506 — 91LD – Abchar 700* – Carbon Cloth
- Data Sheet No. 516/507 — p-Phenylphenol phenol formaldehyde – Abchar 700* – Carbon Cloth
- Data Sheet No. 517/508 — DEN438 – Abchar 700* – Carbon Cloth

Refer to ROCKET NOZZLES, Data Sheet Nos. 509, 510 and 511.

LAMINATED SQUARES

- Data Sheet No. 379/394 — Polybenzimidazole (Imidite 2803 or AFR-151)–Carbon Cloth

Refer to HOT GAS FLOW SPECIMENS, Data Sheet No. 378/394.

- Data Sheet No. 498/488 — Polyphenylene Oxide – Graphite Cloth

Refer to HOT GAS FLOW SPECIMENS, Data Sheet No. 497/488.

*Information on this resin is given on page 39.

PELLET SPECIMENS

- Data Sheet No. 377/394 — Polybenzimidazole (Imidite 2803 or AFR-151)–Carbon Cloth

Refer to HOT GAS FLOW SPECIMENS, Data Sheet No. 378/394.

- Data Sheet No. 391 — 91LD – Pyrolytic Graphite No. 14 Fibers

Pyrolytic graphite No. 14 fibers were cut into approximately 1-inch lengths and dried for 2 hours at 240°F. The dried weight of fibers was 23.4 grams. A resin solution was prepared consisting of 20.1 grams of 91LD and 140 grams of acetone. The dried fibers were soaked in the solution for 60 minutes, then spread on aluminum foil to dry. After a 60 minute air dry, the fibers were vacuum dried for 7 hours.

Following oven drying and "B" staging, the prepreg was molded for 2 hours in the 2-inch disc mold at 300°F and 200 psi pressure. This pressure was used in an attempt to minimize breakage of fibers during molding. However, this low pressure combined with the jack straw arrangement of the impregnated fibers resulted in a porous part. The pellets machined from this molding were honeycombed with small voids.

After a telephone conference, the AFML and Hughes project engineers decided to impregnate the specimens to fill these voids. 91LD resin is unsatisfactory for impregnation because of its high solvent content and the volatiles given off during cure. DEN 438 was therefore chosen as the impregnating resin. The pellets were impregnated three times to obtain a stable weight.

The percentage of voids in the resin matrix filled with DEN 438 was calculated for each pellet as follows:

1. The volume of each machined pellet was calculated from the accurately measured diameter and thickness.

2. The volume of each component was found by

$$\text{Vol}_{91LD} = \frac{\text{Wt}_{91LD}}{D_{91LD}} = \frac{\text{Wt}_{91LD}}{1.26}$$

$$\text{Vol}_{\text{Fibers}} = \frac{\text{Wt}_{\text{Fibers}}}{D_{\text{Fibers}}} = \frac{\text{Wt}_{\text{Fibers}}}{2.07^*}$$

*Density is converted from specific gravity obtained by air and water method.

3. Therefore the volume of voids for each pellet was

$$Vol_{Voids} = Vol_{Pellet} - Vol_{91LD} - Vol_{Fibers}$$

4. The amount of impregnating DEN 438 was calculated by

$$Wt_{DEN\ 438} = Wt_{Pellet\ after\ Impreg} - Wt_{Pellet\ before\ Impreg}$$

5. The volume of DEN 438 impregnated in each pellet was

$$Vol_{DEN\ 438} = \frac{Wt_{DEN\ 438}}{D_{DEN\ 438}} = \frac{Wt_{DEN\ 438}}{1.247}$$

6. The percentage of voids filled with DEN 438 resin in each pellet was

$$\%\ Voids\ filled\ with\ DEN\ 438 = \frac{Vol_{DEN\ 438}}{Vol\ of\ Voids\ before\ Impreg} \times 100$$

The percent voids remaining after impregnation was negative for Specimen No. 1 since the sum of the volumes of 91LD, DEN 438 and fibers was greater than the pellet volume. This negative value results from using the resin content of the 2-inch diameter molding for that of the 3/4-inch diameter pellet. The larger disc was not homogeneous and each pellet probably contained a slightly different amount of resin. Individual resin contents could not be determined without destroying the specimens.

- Data Sheet No. 397b — GR-1 – Carbon Cloth

Refer to ROCKET NOZZLES, Data Sheet No. 397a.

- Data Sheet No. 428 — Abchar 500*–Carbon Cloth
- Data Sheet No. 429 — Abchar 500*–Refrasil Cloth

The Abchar 500* resin pellets were machined from laminates prepared using the dry powder layup technique. The required number of plies of reinforcement were weighed (the carbon cloth was oven dried prior to weighing) and resin added as follows:

- The amount of resin required to give the desired resin content was weighed out.
- The resin weight was divided by the number of plies less one to obtain the amount of resin to be added to each ply.

*Information on this resin is given on page 39.

- This amount was weighed out on an analytical balance and sprinkled uniformly over the first ply of reinforcement.

- More reinforcement and resin were added using this technique until the layup was complete.

- The layup was immobilized by wrapping in aluminum foil prior to molding.

The immobilized plies and resin were transferred to a 600°F press and the resin allowed to melt and flow under pressure. The layup was heated for 2 hours at 300 psi and then slowly cooled under pressure, to room temperature. Two composites, Abchar 500* resin-carbon cloth and Abchar 500* resin-Refrasil cloth were successfully prepared in this manner.

Pellets were machined from these composites using special techniques. The resin is thermoplastic and is similar to a high melting wax. The laminates were cut into 1-inch squares and the pieces chilled in a dry ice-acetone bath (approximately -112°F). This chilling hardened the resin and allowed parts to be made without the resin gumming up from the heat of machining. However, the part had to be sprayed with a can of aerosol coolant periodically while in the lathe. This kept the specimen cold during machining.

The machined parts were still not completely satisfactory. This resin is extremely difficult to machine because of its waxy nature. The surfaces of the specimens were not uniform since the resin did not sufficiently rigidize the reinforcement.

One pellet containing Refrasil cloth was postcured to determine the effect of prolonged high temperature exposure. The specimen was subjected to temperatures ranging from 700° to 900°F over a 48-hour period. This 3/4 inch diameter by 1/2-inch pellet specimen was placed in the 3/4-inch diameter disc mold. The mold had been previously heated to 700°F. The specimen was held at 700°F for 1/2 hour under slightly more than contact pressure. No flow occurred. The mold temperature was raised to 800°F and after approximately 1/2 hour, considerable flow took place. The temperature was dropped to 700°F and the part held there for 18 hours. At the end of this time, the part was again taken to 800°F. No flow occurred. After 8 hours at 800°F, the mold temperature was raised to 900°F. Shortly thereafter, a slight amount of resin flowed from the bottom of the mold. The temperature was lowered back to 800°F and left there overnight. The next morning the mold was reheated to 900°F. This time no flow took place. After 4-1/2 hours at 900°F, the specimen was cooled down to room temperature under pressure.

The specimen appeared unchanged when removed from the mold. Barcol readings before and after high temperature exposure were

*Information on this resin is given on page 39.

negligible (an increase in Barcol is evidence of crosslinking). The specimen had lost 18.5 percent of its original weight. The resin content prior to postcure was 35 weight-percent but only 20.2 weight-percent after postcure.

The remaining pellets were not postcured for two reasons. First, the large weight loss would place the specimens well out of the allowable resin content range. Second, there is insufficient evidence to indicate and appreciable amount of crosslinking took place. To increase crosslinking and consequently improve the high temperature properties is the primary purpose of postcuring.

- Data Sheet No. 431 — Abchar 413*–Para-polyphenylene**–Carbon Cloth

Refer to ROCKET NOZZLES, Data Sheet Nos. 432, 433 and 433b.

- Data Sheet No. 460a — 91LD – Boron Fibers
- Data Sheet No. 460b — 91LD – Boron Fibers

Hughes Aircraft Company received a roll of boron fibers impregnated with 91LD resin. The material was in a 1/8-inch wide flat tape which contained an average of 26 fibers and was one fiber thick. The vendor reported a resin content of 33 weight-percent based on the weight of fibers before and after coating. Six resin extractions with acetone were performed however on pieces randomly selected throughout the roll. The average of the values obtained for the resin content was 31.6 weight-percent.

Pieces of boron fiber tape 4-1/8 inches long were laid side by side to form a square 4-1/8 by 4-1/8 inches in dimensions. The ends of the fibers were held down on a block with masking tape. A second layer 4-1/8 by 4-1/8 inches was laid at right angles over the first and tacked down to the first using a hot tacking iron. A piece of Mylar film was placed on the fibers during the tacking operation to prevent the resin from sticking to the iron. The layup was then cut into sixteen 1 by 1 inch squares with a blanking tool.

Using the blanked plies, two 1 by 1 by 1/2 inch and two 1 by 1 by 3/4 inch moldings were made. The first molding, (1 by 1 by 1/2 inch) was prepared using cure conditions suggested by the vendor. Their recommendations were as follows:

- Place the cut plies in a cold mold.
- "B" stage plies in mold at 200°F for 5 hours after mold reaches temperature.
- Place 200°F mold in 200°F press and apply 200 psi pressure immediately.

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*Information on this resin is given on page 41

**Information on this resin is given on page 40.

- Gradually raise platen temperature to 300°F taking care that pressure on the part does not exceed 200 psi.

- Cure for 2 hours at 200 psi after the mold temperature reaches 300°F.

The resulting molding was porous and while acceptable was not of good quality. The resin had been advanced too far and did not flow. The "B" stage conditions and the initial part of the recommended molding cycle were too long to produce a high quality molding.

Two more parts, one 1/2-inch thick and the other 3/4-inch thick, were molded as follows using less severe "B" staging conditions:

- Cut plies were placed in a cold mold.

- Mold and plies were placed in a 200°F oven for 1 hour.

- Mold was then transferred to a 300°F press and part was molded for 2 hours at 200 psi.

This change in "B" stage conditions resulted in parts having a satisfactory amount of flow. The above processes are shown in Figures 2 through 7.

For the fourth molding, the "B" staging was cut down to 1/2 hour. The resulting 3/4-inch thick part was the best of the four. Upon completion of the last molding, all the parts were postcured using the standard cycle.

Each of the two 1 by 1 by 1/2 inch thick composites were machined into a pellet specimen with plies perpendicular to the axis. The two 1 by 1 by 3/4 inch thick moldings were each machined into a pellet specimen with alternate plies parallel and perpendicular to the axis.

The resin content of the composites were calculated by the standard method, using the average boron fiber content of the prepreg.

Data Sheet No. 465 DEN 438 - Boron Nitride Fibers

One pellet was molded in the 3/4 inch diameter mold from each of the three batches of boron nitride fibers received from AFML. The prepreg was prepared by first cutting the fibers into 1/2-inch lengths and drying them for 2 hours at 220°F. The impregnating solution was prepared by blending 100 pbw DEN 438 resin, 100 pbw methyl nadic anhydride and 1 pbw benzyldimethylamine. The catalyzed resin was diluted with acetone to coating consistency and then evacuated to remove all air. A weighed amount of cut fibers were put into a small beaker and placed in a vacuum chamber. Thirty minutes after air had been removed from the chamber, the resin solution was added into the beaker by a separatory funnel. The fibers were soaked for 30 minutes under vacuum. The excess resin was

Figure 2. Cross-plying 91LD impregnated boron fiber tape to form a large square double ply. (HAC Photo No. R109186)

Figure 3. Tacking double ply with hot iron. Mylar film prevents resin from sticking to iron. (HAC Photo No. R109188)

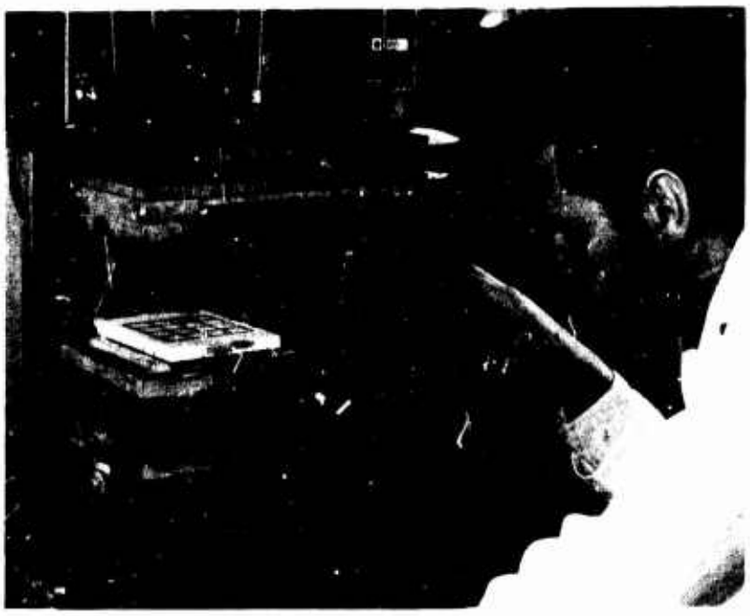

Figure 4. Blanking large double ply into 16 one-inch squares prior to molding. (HAC Photo No. R109189)

Figure 5. Steel rule blanking tool and blanked plies. (HAC Photo No. R109190)

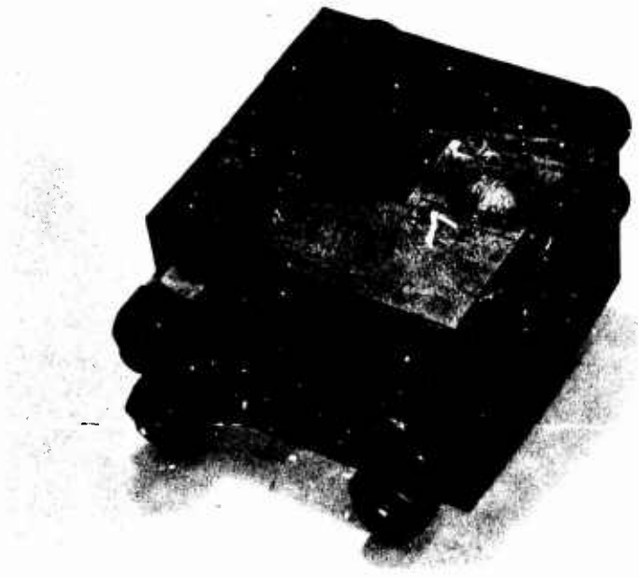

Figure 6. 91LD resin-boron fiber plies stacked in one-inch square mold prior to molding. (HAC Photo No. R109192)

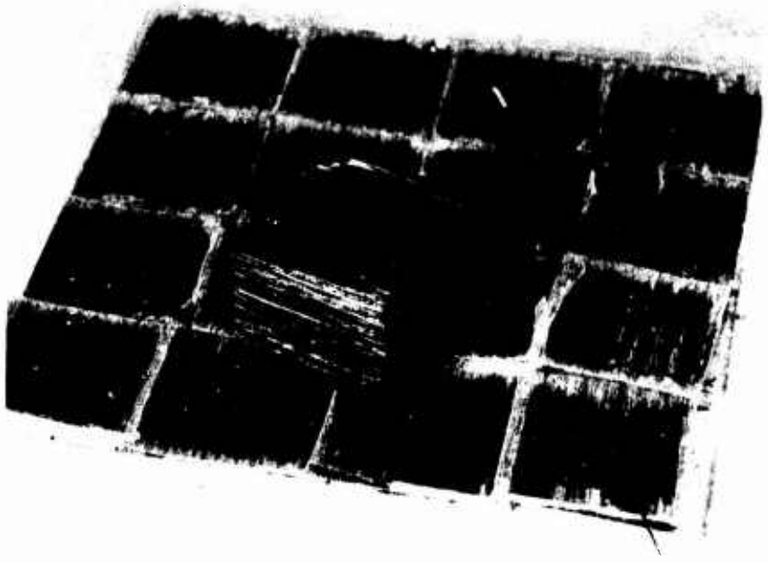

Figure 7. Molded 91LD resin-boron fiber block and blanked plies. One corner of block was removed to determine the porosity of the molding. (HAC Photo No. R109191)

then removed by transferring the fibers to a Buchner funnel and filtering off the remaining solution. The prepreg was air dried, oven dried, "B" staged and molded to produce a pellet within the required resin content tolerance.

- Data Sheet No. 499/488 — Polyphenylene Oxide –Graphite Cloth

Refer to HOT GAS FLOW SPECIMENS, Data Sheet No. 497/488.

- Data Sheet No. 512/506 — 91LD – Abchar 700*– Carbon Cloth
- Data Sheet No. 513/507 — p-Phenylphenol phenol formaldehyde – Abchar 700*– Carbon Cloth
- Data Sheet No. 514/508 — DEN 438 – Abchar 700*–Carbon Cloth

Refer to ROCKET NOZZLES, Data Sheet Nos. 509, 510 and 511.

ROCKET NOZZLES

- Data Sheet No. 397a — GR-1 – Carbon Cloth

GR-1 resin was received in powder form from AFML. The manufacturer recommended the following composition for the impregnating solution:

Material	PBW
GR-1 Resin (Organo Phosphonitrilic)	22.4
Hexamethylenetetramine	2.24
Magnesium Hydroxide	3.21
Ethyl Alcohol	72.1

Additionally, the manufacturer recommended that the magnesium hydroxide be freshly precipitated and added as a slurry in ethyl alcohol. The amount of alcohol added with the slurry was to be considered as part of the total amount in the formulation.

In the initial experimental moldings, reagent grade magnesium hydroxide was used to determine the feasibility of its substitution for freshly precipitated material. Several 3/4-inch diameter pellets were molded using various conditions but none of these were satisfactory. These results indicate that reagent grade material cannot be substituted for freshly prepared hydroxide.

*Information on this resin is given on page 39.

Freshly precipitated magnesium hydroxide was made from a magnesium acetate solution and sodium hydroxide using the method recommended by the vendor. This method is as follows:

- $Mg(OH)_2$ was precipitated by adding excess sodium hydroxide to an aqueous magnesium acetate solution.
- The $Mg(OH)_2$ gel was washed thoroughly with water until excess base was removed. Centrifuging was necessary because the gel tends to hold water.
- The material was given a final wash with absolute ethyl alcohol to remove the last traces of water.
- The wash alcohol was removed by centrifuging and more ethyl alcohol was added to the $Mg(OH)_2$ paste to make a thick slurry. The weight percent alcohol in the slurry was determined by drying a weighed sample to a constant weight and calculating the percent weight loss.

A second batch of resin was compounded using the freshly precipitated magnesium hydroxide. A pellet was successfully molded from carbon cloth impregnated with this resin batch.

The following technique was used to compound GR-1 resin impregnating solution. First, a weighed amount of resin powder was combined with ethyl alcohol for 5 minutes in a Waring Blendor. The magnesium hydroxide alcohol slurry was added and blended into the solution. The amount of alcohol added from the slurry combined with the initial weight of alcohol used, made up the required weight percent of solvent. Finally, a weighed amount of hexamethylenetetramine was blended into the mixture prior to coating.

Using general procedures, dried carbon cloth was spatula coated with the impregnating mixture. The coated cloth was air-dried for 2 hours and vacuum-dried for 16 hours. Although the vendor recommended a 10 to 20 hour "B" stage at 200 to 220°F under vacuum, this length of time was found to be excessive. An experimental laminate made with material "B" staged 10 hours at 200°F did not fuse. No resin flow took place because the prepreg had been advanced too far. Prepreg "B" staged under vacuum was found to attain a stable weight after 6 hours. Another experimental laminate made with this prepreg was satisfactory.

Using the laminating fixture, a 5 by 3 by 3 inch laminate was successfully molded with prepreg "B" staged 6 hours. However, before postcuring this block, a 2-inch diameter by 1/2-inch disc molded from the same materials was postcured using the cycle recommended by the vendor. This disc "blew-up" in postcure. After consulting with the AFML Project Engineer, a postcure study was made. This study revealed that a thick composite will not "blow-up" if a slow temperature

rise is used. As an additional safeguard, the AFML Project Engineer recommended oversized nozzle blanks be machined from the molded composite before postcure. Also, he recommended drilling the pilot hole in each blank to allow volatiles to escape more easily. Two drilled nozzle blanks were then successfully postcured with an extended schedule.

During the machining of the first rocket nozzle insert, the post-cured cylindrical blank broke into several pieces. The GR-1 resin appeared to have poor interlaminar shear strength and the part broke as it was being machined to the outside dimension.

The remaining oversized cylinder was successfully machined into an ASD No. 4 nozzle. Additionally, three 3/4-inch diameter by 1/2-inch pellet specimens were machined from pieces of the first cylinder. These pellets contain 40 weight-percent resin and although not requested by AFML, were shipped for whatever value they may have.

- Data Sheet 403b — Skybond 700 (Polyimide)–Carbon Cloth

A 4 by 2-1/2 by 2-1/2 inch Skybond 700 – carbon cloth CCA-1 block was molded using the laminating fixture. The prepreg contained 56.2 weight-percent resin before molding to compensate for the large resin flow and volatile loss during molding. Additionally, the plies were vacuum dried for 16 hours before "B" staging to remove the remaining N-methyl 2-pyrrolidone (NMP). A small amount of this high boiling point solvent can cause "blow-up" in a thick laminate. After molding, the block was cut in half prior to postcure to minimize the possibility of "blow-up." After postcure, only one of the pieces was successfully machined into a nozzle insert. This insert was bonded into a housing and shipped.

- Data Sheet No. 432 — Abchar 413*–Para-polyphenylene*–Carbon Cloth
- Data Sheet No. 433 — Abchar 413*–Para-polyphenylene**–Refrasil Cloth
- Data Sheet No. 433b — Abchar 413*– Para-polyphenylene*–Refrasil Cloth

A Waring Blendor was used to intimately mix the Abchar 413* solution and the para-polyphenylene powder. The materials were blended for 15 minutes into a fine dispersion which was used for dip coating the carbon and Refrasil cloths. This blending process broke down the particles of para-polyphenylene fine enough for the filler to be deposited in the interstices of the cloth. Failure to do this, results in the powder flaking off the cloth during the blanking of plies.

The para-polyphenylene was considered a filler in determining the resin content of the moldings. During impregnation, known weights

*Information on this resin is given on page 41.
**Information on this resin is given on page 40.

of para-polyphenylene were added to the reinforcements. Therefore the resin content could be determined after molding by subtracting the weight-percent of cloth and filler from 100 percent. The resin content after postcure could not be determined however. At the height of the postcure cycle, 550°F, volatiles are lost from the para-polyphenylene as well as from Abchar 413. There is no way to determine the amount of volatiles lost by each component. Therefore only the weight-percent of reinforcement after postcure was reported.

Three Refrasil cloth nozzles, two carbon cloth nozzles and one carbon cloth, 3-1/2-inch diameter disc were prepared with the Abchar 413 – para-polyphenylene mixture. Two of the Refrasil cloth nozzles contained 15 weight-percent para-polyphenylene; the third contained 20 weight-percent. The carbon cloth nozzles and 3-1/2-inch diameter disc contained 15 weight-percent of the para-polyphenylene.

The two 15 weight-percent Refrasil cloth nozzles and the carbon cloth nozzles were successfully machined and bonded into nozzle housings. However the carbon cloth 3-1/2-inch diameter disc tended to delaminate badly during machining. Carbon cloth has poor wetting qualities and when a large weight of filler is added to the impregnating resin, poor interlaminar shear properties result. Only two satisfactory 3/4-inch diameter pellets were machined from the disc.

The Refrasil cloth nozzle containing 20 weight-percent para-polyphenylene also had poor interlaminar shear strength. Special precautions were therefore taken to prevent breakage of the nozzle during machining. A nozzle housing was modified to allow its use as a machining fixture by trimming it to a length of 2.06 inches and removing the flange. The step in the nozzle insert was then machined and the insert bonded into the short housing with polyglycol. The remaining dimensions of the insert were machined with the possibility of delamination minimized. Upon completion of machining, the housing and insert were heated for 1 hour in a 275°F oven. At 275°F, the polyglycol melted and the insert was easily pushed out of the special housing. The insert was carefully washed in hot water to remove the remaining traces of polyglycol and dried for 2 hours in a 225°F oven. Finally the insert was bonded into a regular nozzle housing using standard procedures.

However, the nozzle insert was short in some places up to 0.040 inch because edges of the stepped end had crumbled during machining. One end of a 1.173-inch diameter teflon plug was tapered to the 20 degree angle of the nozzle insert. After insertion of this plug, adhesive was forced into the gap to fill out the broken edges of the insert. The adhesive in the housing was cured and a light skin cut over the surface of the housing flange removed any excess bonding material.

- Data Sheet No. 466a — DEN 438 – Boron Fibers
- Data Sheet No. 466b — DEN 438 – Boron Fibers

Fabrication Method

AFML requested two rocket nozzles reinforced with boron fiber. The resin matrix for these specimens was DEN 438 epoxy novolac resin. AFML supplied two shipments of coated fibers in the form of 1-3/4-inch wide strips, one fiber thick. The impregnated pieces of boron fiber tape were laid side by side to form a square. Four 7-1/8-inch lengths of 1-3/4-inch wide tape were cross plied with four similar pieces and the layup tacked down with a tacking iron. The prepreg was blanked into 16 pieces 1-3/4 by 1-3/4 inches using a blanking die and press. Each piece contained two plies with the fibers in one ply being perpendicular to the fibers in the other. The layup of the rocket nozzle blanks, therefore, consisted of plies laid perpendicular to the nozzle axis with adjacent plies perpendicular to each other.

During cure of the first nozzle blank, a large volume of resin flowed from the mold.* The resulting molding was cracked when removed from the mold. This molded block was twice impregnated with additional DEN 438 as follows:

- The impregnating resin was prepared by blending 100 pbw DEN 438, 100 pbw methyl nadic anhydride and 1 pbw benzyldimethylamine.
- Air was removed from the mixture under vacuum.
- The block was placed in a container and put under vacuum for 1/2 hour.
- The catalyzed resin was added to the container, under vacuum, until the block was immersed. The molding was then soaked under vacuum for an hour.
- The container with the block immersed in resin was removed from the vacuum chamber and placed in a pressure pot for 1 hour at 500 psi.
- The molding was removed from the pressure pot and excess resin removed by wiping with an acetone-soaked tissue.
- The impregnated molding was placed back into the 1-3/4 by 1-3/4 inch square mold. The part was cured at 500 psi pressure and 300°F for 1 hour during the first cycle and overnight during the second cycle.

*The first nozzle blank consisted of prepreg from the first AFML shipment only.

X-rays of the block showed all cracks to be filled with resin after the second impregnation. The molding was then postcured in an oven using the fast postcure recently instituted for DEN 438 (16 hours room temperature to 275°F, 17 hours at 275°F, 6 hours from 275° to 400°F, 1 hour at 400°F. However, after completing this schedule, the block was again cracked. The boron fiber molding was reimpregnated with DEN 438 for a third and fourth time. When another X-ray revealed the cracks were finally filled, the part was placed in the shop for machining.

Some problems were encountered in the early stages of machining the composite. The first problem was the drilling of the pilot hole. This was finally solved by using a Felker Die-Mitt drill holder with a 3/8-inch diamond core drill. This drill holder permits a flow of water through the drill during the machining operation keeping both the drill and part cool. Dust from the drilling operation is also washed away. The remaining dimensions were successfully machined in a lathe equipped with a tool post grinder. It was not possible to machine this material with other than a rapidly rotating diamond tool.

The second nozzle blank was made using primarily the second shipment of boron fibers. The balance of the required plies were supplied from the first shipment. Very little flow occurred during molding but this flow was sufficient for the plies to fuse satisfactorily.

X-rays of the block, however, revealed a possible weak area in the molding. The part was therefore impregnated with additional DEN 438 using vacuum followed by high pressure. The resin was cured by putting the block back in the mold and applying heat and pressure.

After impregnation, the part was postcured. The block was then sent to the shop for machining but broke in two during the first operation Each piece was impregnated with DEN 438 and put back into the mold together for cure under pressure. After cure, the bonded block was successfully machined to the ASD No. 4 configuration. Figure 8 illustrates the machined nozzle insert prior to bonding.

Calculation of Resin Content

Because of the cost and time involved, no resin extractions were run on the impregnated DEN 438 boron fiber tape. For the same reasons, no quantitative determination was made to find the amount of boron and tungsten present in the molded composites. Resin contents were estimated using the density value for a 4 mil boron fiber given in Technical Report AFML-TR-65-21, "Boron Filaments and Composites, March 1965." The 4-mil diameter was selected as being the average fiber diameter based on measurements of previously extracted fibers. The density of this fiber was 2.625 gms/cc as interpolated from Figure II-3 of AFML-TR-65-21.

Figure 8. ASD No. 4 rocket nozzle insert machined from a DEN 438 resin-boron fiber composite. (HAC Photo No. R110841)

The weight of boron fibers in the DEN 438-boron fiber nozzle blanks was calculated as follows:

- Volume of a single fiber was calculated from the length and the diameter of the fiber.
- Volume of all fibers in a ply was determined by multiplying the single fiber volume by the number of fibers in a ply.
- Volume of boron fibers in composite was then found by multiplying the volume of fibers in a ply by the number of plies in the molding.
- The weight of boron fibers was finally calculated using the formula Weight = Density × Volume.

Using this calculated weight, the resin content of each composite was calculated from the boron fiber weight and the total weight of the composite. Each resin content value was used to calculate the effective resin density for use in determining the volume percent voids in the resin. In all cases, this effective resin density was found to be higher than the measured pure resin density. This is an impossibility. Obviously, the assumed values for volume and density of the boron fibers were highly inaccurate.

Resin content values corresponding to a range in boron fiber diameters were calculated. These values were determined under the assumption that the nozzles were voidfree and contained only boron fibers and DEN 438 resin.

The upper limit of the range was calculated by assuming all fibers were 3 mils in diameter, the lower that all were 6 mils. A resin content was also calculated which corresponded to the average fiber diameter of 4 mils.

Using the following formula, for a 100-gram composite:

$$D_C = \frac{100}{\frac{W_R}{D_R} + \frac{100-W_R}{D_{BF}}}$$

then

$$W_R = \frac{100\, D_R\, (D_{BF} - D_C)}{D_C\, (D_{BF} - D_R)}$$

where

W_R = Weight-percent of resin

D_{BF} = Density of boron fibers

upper limit (3 mil diameter fibers)	2.88 gm/cc
average (4 mil diameter fibers)	2.62 gm/cc
lower limit (6 mil diameter fibers)	2.46 gm/cc

D_C = Density of composite

No. 1 nozzle	2.03 gm/cc
No. 2 nozzle	1.87 gm/cc

D_R = Density of resin 1.247 gm/cc

	No. 1 Nozzle	No. 2 Nozzle
Resin content – upper limit	32.0 wt-%	41.2 wt-%
Resin content – 4 mil fiber diameter	26.5 wt-%	36.5 wt-%
Resin content – lower limit	21.8 wt-%	32.4 wt-%

Possible explanations for the high values of resin content obtained for the second nozzle are given below:

- The lower density of the No. 2 nozzle indicates the specimen may not be void free.
- Unlike the molding of No. 1 nozzle, little resin flow took place in molding No. 2 nozzle. The resin content of the machined specimen would be higher and the density lower with more resin retained in the molding.

• Data Sheet No. 509	91LD – Abchar 700* – Carbon Cloth
• Data Sheet No. 510	p-Phenylphenol phenol formaldehyde – Abchar 700*–Carbon Cloth
• Data Sheet No. 511	DEN 438 – Abchar 700* – Carbon Cloth

Several specimens were fabricated using Abchar 700* powder as a filler and carbon cloth CCA-1 as reinforcement. Three resin systems were used and three types of specimens fabricated from each resin system. These systems consisted of 91LD resin, p-phenylphenol phenol

*Information on this resin is given on page 39.

formaldehyde resin and DEN 438 resin. The types of specimens prepared were rocket nozzles and 8 by 6-1/2 by 1/2-inch laminates which were machined into pellets and hot gas flow, type D specimens (3-1/2 by 2 by 1,) inches).

The fabrication techniques were similar regardless of the resin system. The carbon cloth was oven dried to remove residual moisture and then weighed. The weight of Abchar 700 powder required was calculated using the ratio of 15 pbw filler to 55 pbw of cloth. The final desired resin content was 30 weight-percent. Therefore, the weight of resin varnish was calculated to contain in resin solids, twice the weight of Abchar 700* filler.

A Waring Blendor was used to intimately mix Abchar 700* into the resin solution. The materials were blended for 15 minutes after which acetone was added to thin the mix to coating consistency. This blending process broke down the particles of filler fine enough for the filler to be deposited in the interstices of the cloth. Failure to do this, results in the powder flaking off the cloth during the blanking of plies.

The carbon cloth was spatula coated, air dried, oven dried, 'B' staged, molded and postcured using the conditions indicated in the tables under Fabrication Details. The resin and filler content for the cured and postcured specimens were calculated as indicated below.

1. A known weight of cloth was uniformly coated with an exact amount of resin-filler blend containing a known weight of filler.

2. The resin content was found at each stage of the prepreg's preparation by first finding the weight percent of reinforcement and filler. The sum of these components when subtracted from 100 percent gave the weight-percent of resin.

3. New weights of reinforcement and filler were calculated after the prepreg was cut or blanked into plies. The weight-percentages of each component was multiplied by the charge weight of the prepreg.

4. The resin contents after molding and postcure were found using the procedures in Step No. 2.

The volume-percent voids in the resin matrix was calculated from the following formulas.

1. $$D_{ML} = \frac{\Sigma W_{ML}}{\Sigma V_{ML}} = \frac{\Sigma W_{ML}}{V_{VFR} + V_F}$$

*Information on this resin is given on page 39.

since

$$V = \frac{W}{D}$$

then

$$\approx \quad D_{ML} = \frac{\Sigma W_{ML}}{\frac{W_R}{D_{VFR}} + \frac{W_C}{D_C} + \frac{W_F}{D_F}}$$

where

D_{ML} = Density of molding or laminate

W_{ML} = Total weight of the components in the molding or laminate

V_{ML} = Total volume of the components in the molding or laminate

V_{VFR} = Volume of the void filled resin

V_C = Volume of cloth

V_F = Volume of filler

D_{VFR} = Density of void filled resin

D_C = Density of cloth

D_F = Density of filler

W_R = Weight of resin

W_C = Weight of cloth

W_F = Weight of filler

2. Volume percent voids in resin = $\frac{D_R - D_{VFR}}{D_R} \times 100$

Calculating the final resin, reinforcement and filler content as well as the volume-percent of voids in the resin matrix was contingent on one factor. This factor was whether the Abchar 700* filler lost any weight during postcure. To determine this, a series of pellets was molded containing either pure resin or resin and filler in a 2:1 mixture (30 pbw: 15 pbw). All three resin systems were used and all members of a series were molded under identical conditions.

The resins were dried in an oven to remove any solvent and then B-staged until they formed brittle solids. These solids were then pulverized into powders which were blended with Abchar 700.* All molded pellets were weighed before and after postcure.

The average weight-percent lost during postcure was calculated for each of the three pure resins. These values were fairly uniform for a given resin. For the composites, an assumption was made as follows.

$$\text{Wt Loss}_{\text{Comp.}} = \text{Percent Wt Loss}_{\text{Resin}} \times \text{Wt Frac. Resin} + \text{Percent Wt Loss}_{\text{Filler}} \times \text{Wt Frac. Filler}$$

The percent-weight loss of filler can be found since all other components of the equation are known.

In 91LD and p-phenylphenol phenol formaldehyde resins, all the weight loss of the composite was attributable to the resin only. The Abchar 700* when encapsulated in these resin matrices does not lose weight when po cured using the standard postcure (72 hours from 275° to 400°F, 4 hours at 400°F).

DEN 438 resin also does not lose more than 0.1 to 0.2 percent weight under these conditions. However, the DEN 438 – Abchar 700* composites lost several percent during postcure. Additionally, DEN 438 - Abchar 700* nozzles when removed from postcure had expanded between 20 to 30 percent in height. Consultation with Dr. N. Bilow revealed that either the anhydride from the MNA catalyst or groups on the epoxy novolac resin could have reacted with the Abchar 700.*

Both reactions may even have taken place with the possible liberation of CO_2. Evidence indicates that DEN 438 softens at 400°F and, therefore, would have expanded if CO_2 was given off at that time. Evolution of CO_2 would also explain the weight loss of the resin-filler composite.

Dr. Bilow further stated that DEN 438 – Abchar 700* composites are probably improperly cured since this reaction changes the stoichiometric proportions of the resin and catalyst. However, a long detailed program would be required to determine what percentages of each component is necessary to give a proper cure.

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*Information on this resin is given on page 39.

To prevent this foaming of DEN 438—Abchar 700* Nozzles during postcure, new nozzles were molded which were cured and postcured in the press under high pressure. These parts were cooled under pressure to below 200°F before removal from the press. Thus, the nozzle blanks were well under the softening point of the resin and were of satisfactory quality. The weight-percent of resin and filler shown in Table 5 and in Data Sheet No. 511 are approximate. There was a slight weight loss during molding. This weight loss cannot be accurately distributed between the resin and filler, however.

*Information on this resin is given on page 39.

UNSUCCESSFUL EXPERIMENTS

Two GR-1 resin-Refrasil cloth C100-48 nozzles were also requested by AFML. A 5 by 3 by 3 inch block was successfully molded from GR-1-Refrasil prepreg using the laminating fixture. This prepreg had been prepared identically to the previous GR-1 carbon cloth material.* The block was cut in half and each piece was to be rough machined into two oversized nozzle blanks. Again each cylinder would contain a pilot hole to allow volatiles to escape more easily. During machining however, each part broke into several pieces although special precautions were taken to prevent this. These precautions proved to no avail since the composite had virtually no interlaminar shear strength. A new blank was not molded since the remaining GR-1 resin was used to prepare this blank.

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*See Specific Specimen Preparation, Rocket Nozzles, Data Sheet No. 397a.

MATERIAL STUDIES

DEN 438 Resin Postcure

A considerable cracking problem has resulted when attempting to postcure large composites of DEN 438 resin. The standard B-1 postcure with its slow 72-hour rise to 400°F invariably results in cracked nozzle blanks and 2-inch diameter discs. The faster 6-hour temperature rise postcure is at best marginal. The origin of the problem seems to be that DEN 438 resin softens and weakens in this upper temperature range. This allows any entrapped volatiles to "blow" the part as they seek to escape. As further evidence, two DEN 438 – Abchar 700*– carbon cloth nozzle blanks actually increased over 1 inch in height during postcure. A reaction between components resulted in an expansion of the resin matrix between the plies similar to foaming. This could not have taken place if the resin remained rigid.

DEN 438 resin loses only a negligible amount of its weight during postcure. However the AFML Project Engineer reports that test results between postcured and unpostcured DEN 438 resin show that the former is superior. Therefore postcuring DEN 438 remains highly desirable.

A new technique was instituted towards the end of the report period. This technique combines the curing and postcuring of the part. Programmed controllers are attached to the press platens and the molding and postcure temperature cycle are combined. The part remains under pressure during the entire cure and postcure cycle. Two DEN 438 resin – Abchar 700*–carbon cloth nozzles were remade successfully using this method. This combination of a programmed cure-postcure will be used on all DEN 438 parts to be made in the future.

*Information on this resin is given on page 39.

Polyaminoborane

Polyaminoborane resin was received from AFML for evaluation. A thermogravimetric analysis (TGA) was run on this white powder. The results of the TGA are shown in Figure 9. The sample decomposed at 160°C and the weight of the residue thereafter remained constant.

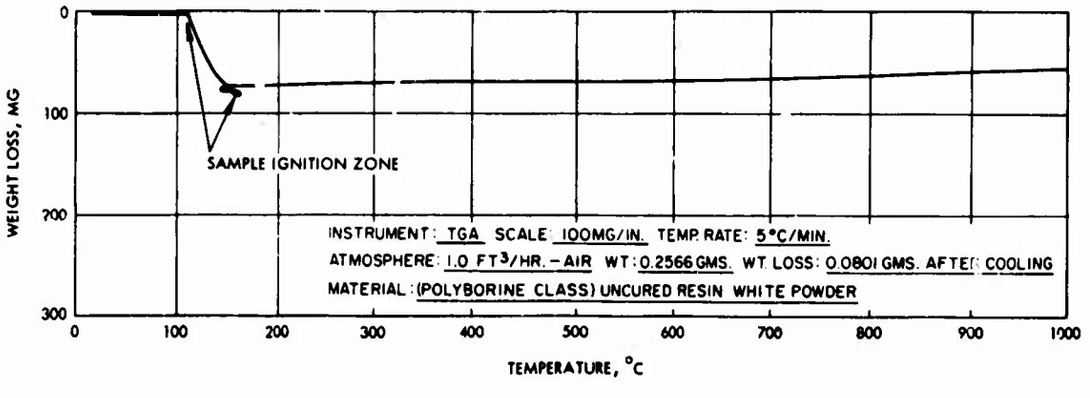

Figure 9. Thermogravimetric analysis of polyamino borane resin.

Ferrocenylenes

Seven ferrocenylene resins were received from AFML for evaluation. AFML requested one 1-1/8-inch diameter by 1/4-inch solid resin slug and one 3/4-inch diameter by 1/2-inch reinforced pellet from each resin type, if possible. Small moldings, 3/4-inch diameter by 1/16-inch were prepared to conserve material while developing the proper molding cycle. The following table lists a summary of the results.

Ferrocenylene Designation	3/4-inch Diameter by 1/16-inch Resin Slug	1-1/8-inch Diameter by 1/4-inch Resin Slug	3/4-inch Diameter Reinforced Composite
HR-1	Several attempts were made to mold parts at different temperatures. A part was successfully molded at 450°F.	A part was molded at 450°F but broke when removed from the mold.	A lacquer was made using benzene as solvent. Sixteen 3/4-inch discs of carbon cloth CCA-1 were dip coated six times to obtain approximately 40 weight-percent resin content. The plies were molded for 45 minutes at 450°F. Plies did not bond together.
HR-2	Several attempts were made to mold parts at different temperatures. One part was molded at 600°F and 10,000 psi for 60 minutes. This part had only a small amount of cracking.	A part was molded at 600°F and 10,000 psi for 60 minutes. Small cracking took place when backing on both sides of the molding was removed. However the part broke into several pieces on standing.	No attempt was made to mold a reinforced composite.
HR-3	Several attempts were made to mold parts at different temperatures and pressure. A successful molding was prepared curing at 530°F and 20,000 psi.	A part was molded at 530°F and 20,000 psi. However one Teflon disc used as a backing flowed and the resulting molding was uneven in thickness.	No attempt was made to mold a reinforced composite.
HR-4	A good molding was prepared in the second attempt. The part was molded at 500°F and 20,000 psi for 20 minutes.	A part was molded at 500°F and 20,000 psi for 20 minutes.	Sufficient material remains to mold a composite.
HR-5	On third attempt, a solid part was molded using 500°F and 20,000 psi.	A part was molded at 500°F and 20,000 psi.	No attempt was made to mold a reinforced composite.
HR-6	A good molding was prepared in the first attempt. The part was molded at 530°F and 20,000 psi for 20 minutes.	An attempt was made to mold a part at 530°F and 20,000 psi. Almost all the resin flowed out of the mold. Only a thin film was left.	No attempt was made to mold a reinforced composite.
HR-7	Several attempts were made to mold this material using various pressures and cure temperatures. All proved unsuccessful since the parts remained in powder form or broke when removed from the mold.	No attempt was made to mold a 1-1/8-inch diameter resin slug.	No attempt was made to mold a reinforced composite.

MISCELLANEOUS NOTES

- Data Sheet No. 405 — DEN 438 – Refrasil Cloth Nozzle

Nozzle No. K-63-1 was 0.025 inch short of the required 2.062-inch dimension. The 0.375-inch step was within tolerance however. A fillet of Hapex 1208 adhesive was added to the short end after the nozzle was bonded in the housing. This fillet was then ground to the required angle.

- Data Sheet No. 501 — F-170 (Imide) – Carbon Cloth Hot Gas Flow Specimens
- Data Sheet No. 502 — F171 (Polyarylene Phenolic) – Carbon Cloth Hot Gas Flow Specimens
- Data Sheet No. 503 — F172 (Polyphenylene Phenolic) – Carbon Cloth Hot Gas Flow Specimens

The laminates containing F170, F171, and F172 were cured and postcured using the vendor's recommendations. Additionally, the prepregs containing F170 and Abchar 413 resins were vacuum-dried for 16 hours at room temperature just prior to molding. Vacuum drying was necessary to remove volatile constituents which could cause "blow-up" during cure and postcure of these laminates.

APPENDIX

FORMULATIONS USED WITH MULTIPART RESIN SYSTEMS

Material	Parts by Weight
DEN 438	
DEN 438 (Epoxy Novolac)	100
Methyl Nadic Anhydride	100
Benzyldimethylamine	1
GR-1	
GR-1 (Organo Phosphonitrilic) Resin	22.4
Hexamethylenetetramine	2.24
Magnesium Hydroxide	3.21
Ethyl Alcohol	72.1
PH990	
PH990 (Organo Phosphonitrilic) Resin	100
Hexamethylenetetramine	10
Magnesium Oxide	10

RESIN SYNTHESES

All resins were synthesized by the Chemical Synthesis Group of the Materials Technology Department under the direction of Dr. Norman Bilow.

p-Phenylphenol Phenol Formaldehyde

Batch No. B2353-40B

318g p-phenylphenol
75 g NaOH in 330 ml water
100 ml methanol
228 g formalin (37 percent)

The above reactants were mixed and heated at 70°C for 8 hours. The solution was then acidified and the precipitated resin was thoroughly washed and vacuum dried at 60°C. The resin was then dissolved in a mixture of acetone, benzene tetrahydrofuran and ethanol and after filtering was blended with 1112 g of a 64 percent solids content phenolic prepolymer (CTL 91LD). The combined mix was heated to 99°C for about 1 hour and excess solvent was removed to yield 1370 g of lacquer having 78 percent solids content.

Abchar 220

2,7-Dihydroxynaphthalene Phenol Formaldehyde with High Barium Content Catalyst

Batch No. B2353-61B

2,7-Dihydroxynaphthalene (180 g), phenol (317 g) and formalin 508 g, 37 percent CH_2O, inhibited) were stirred together and barium hydroxide monohydrate (42 g) was added. The solution was heated to 70°C and maintained at this temperature for 2 hours. After cooling, the pH was adjusted to 7. Water was removed under vacuum while maintaining the temperature below 70°C. The dried viscous resin was diluted with absolute ethanol to yield a lacquer containing approximately 70 percent solids.

Abchar 500

High Molecular Weight Polyphenylene

Batch No. C2426-11B

One mole of biphenyl, one mole of meta terphenyl, four moles of cupric chloride and 2 moles of aluminum chloride were reacted together for 3 hours at 100°C. The crude reaction product was then meticulously scrubbed with hydrochloric acid and water. After thorough drying the polymer was continuously extracted with boiling chlorobenzene. The insoluble part of the polymer was then continuously extracted with boiling trichlorobenzene. That polymer which dissolved in the trichlorobenzene was collected by solvent removal under heat and vacuum and is the polymer designated C2426-11B.

Abchar 700

Intractable Polyphenylene

Batch No. C2426-15C

Intractable polyphenylene isolated from a cationic oxidative polymerization of an equi-molar mixture of biphenyl and terphenyl was repeatedly washed with concentrated aqueous hydrochloric acid until the wash was virtually colorless. The polymer was then extracted continuously with boiling trichlorobenzene to remove any residual soluble polyphenylene. It was then washed with naphtha and dried with heat and vacuum. This resin is equivalent to Batch C1414-27 of Abchar 700 previously prepared.

Abchar 413

Polyphenylene

Batch No. C2943-58

195 g polyphenylene (92.53 percent C, 4.72 percent H, 2.14 percent Cl, 0.35 percent ash, mp 160-165°C, M.W. 1000) was dissolved in 500 ml of trichloroethylene and heated to reflux until homogeneous. To this solution was then added a standard curing agent solution derived from 65 g xylyleneglycol, 19.5 g p-toluenesulfonic acid monohydrate and 460 ml chloroform. The combined solutions were stirred together at 70°C for 19 hours to yield 1400 g of lacquer with 17 percent solids.

Abchar 412

Polyphenylene

Batch No. C2943-74

Polyphenylene (melting range 150-250°C, M. W. ≃ 1200) was dissolved in 500 ml of trichloroethylene. After heating at reflux for 2 hours, a solution of curing agent was added which had been prepared from p-xylylene glycol (125 g), p-toluene sulfonic acid monohydrate (38 g) and chloroform (900 ml). The curing agent solution was heated at reflux for 20 hours, while removing water, before use. Additionally, the polyphenylene-curing agent mixture was heated at 70°C for 20 hours prior to use. Lacquer yield 1900 g (20 percent solids).

Abchar 413

Polyphenylene

Batch No. C3128-48

263 g of polyphenylene* was slurried in 180 ml tetrachloroethane with 80 ml of trichloroethylene. The slurry was gradually heated to reflux while stirring constantly. After 1 hour at reflux, 500 ml of trichloroethylene was added. One and one-half hours later, the curing agent solution was added, the latter being made from 88 g of xylylene glycol, 29 g of toluenesulfonic acid and 800 ml of chloroform.

The combined solutions were heated at reflux for 20 hours. The final lacquer weighed 2000 g and contained approximately 18 percent solids.

Abchar 600

Para-Polyphenylene

Batch No. C3128-49B

A slurry of aluminum chloride (700 g) in benzene (2000 ml) was heated to reflux and anhydrous cupric chloride (1717 g) was then gradually added with caution. After stirring and refluxing overnight the reaction mix was poured over a mixture of ice and concentrated aqueous HCl. The precipitated polymer was collected by filtration and washed several times, first with concentrated acid and then with water. It was dried using heat and vacuum. The yield was 135 g.

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*Soluble in chlorobenzene and insoluble in a 15 percent benzene – 85 percent hexane mixture. Mol Wt ≃ 1000-1200, Melt Pt 180-220°C.

Abchar 600

Para-Polyphenylene

Batch No. C3128-50

A mixture of benzene (2000 ml) and aluminum chloride (700 g) was heated to gentle reflux. Anhydrous cupric chloride (1719 g) was then added cautiously and in small portions over a 1 hour period. The reaction mixture was then allowed to heat at reflux for approximately 22 hours. After cooling it was treated with a mixture of ice and concentrated aqueous hydrochloric acid. The precipitated polymer was collected by filtration, scrubbed repeatedly with concentrated aqueous HCl, then water. After drying the polymer weighed 135 g.

Abchar 413

Polyphenylene

Batch No. C3128-52

179 g of polyphenylene (chlorobenzene soluble type) was dissolved in trichloroethylene (500 ml) and heated at reflux for 2 to 3 hours. A solution of xylyleneglycol (60 g), p-toluene sulfonic acid mono-hydrate (18 g) and chloroform (600 ml) was added. After refluxing for 20 hours part of the excess chloroform was distilled off. The lacquer weighed 1842 g and contained 13 percent solids.

TABLE 1

PROPERTIES OF HOT GAS FLOW TYPE D[1] SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume Percent Voids in Resin	
378/394	N151-35-C	1.34	70	37.4	62.6	29.7	Imidite 2803 (AFR-151) resin with Carbon cloth CCA-1 as reinforcement
442/434	DDPO-35-GU	1.28	43	37.0	63.0	–	Diphenyl oxide resin with Graphite cloth G1550, uncoated, as reinforcement
445/435	SG7-35-GU	1.17	10	37.4	62.6	–	Skybond 700 resin with Graphite cloth G1550 uncoated, as reinforcement
448/436	CP-35-GU	1.33	45	36.2	63.8	–	Chrome-P resin with Graphite cloth G1550, uncoated, as reinforcement
454/438	DNB-35-GU	1.40	45	38.0	62.0	27.7	2,7 dihydroxynaphthalene phenol formaldehyde resin (high barium content catalyst) with Graphite cloth G1550, uncoated, as reinforcement
494/487	TP(H)-35-GU	1.46	82	32.6	67.4	–	High Tungsten-P resin with Graphite cloth G1550, uncoated, as reinforcement
497/488	PPO-35-GU	1.30	30	35.4	64.6	25.2	Polyphenylene oxide resin with Graphite cloth G1550, uncoated, as reinforcement
500	9-35-C	1.38	70	37.9	62.1	22.3	91LD resin with Carbon cloth CCA-1 as reinforcement
501	170-35-C	1.16	58	33.6	66.4	–	F170 resin with Carbon cloth CCA-1 as reinforcement
502	171-35-C	1.32	62	36.1	63.9	–	F171 resin with Carbon cloth CCA-1 as reinforcement
503	172-35-C	1.32	25	34.9	65.1	–	F172 resin with Carbon cloth CCA-1 as reinforcement
504	PP413-35-C	1.32	45	36.6	63.4	31.8	Abchar 413 resin with Carbon cloth CCA-1 as reinforcement
505	DP4-35-C	1.31	–	37.3	62.7	–	Phenyl aldehyde (DP-4-31) resin with Carbon cloth CCA-1 as reinforcement

*For further identification of materials, see Table 14 on page 66.

TABLE 1 (CONTINUED)

PROPERTIES OF HOT GAS FLOW TYPE D[1] SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
515/506	9-PP700-30-C	1.32	–	30.0	52.6 Reinforcement 14.4 Filler	27.6	91LD resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
516/507	PPP-PP700-30-C	1.29	–	30.9	54.3 Reinforcement 14.8 Filler	29.2	p-Phenylphenol phenol formaldehyde resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
517/508	D-PP700-30-C	1.20	–	29.1[2]	55.7 Reinforcement[2] 15.2 Filler[2]	–	DEN 438 resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement

1 Dimensions for type D Hot Gas Flow specimens are 3.333" ± 0.010" x 2.000" ± 0.010" x 0.502" ± 0.002".

2 The final resin, reinforcement and filler contents could not be determined because of a weight loss due to possible side reactions. Such reactions could take place between the epoxy resin and Abchar 700 or the epoxy resin catalyst and Abchar 700 or both. The values reported are for the laminate as molded.

*For further identification of materials, see Table 14 on page 66.

TABLE 2

PROPERTIES OF LAMINATES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
363	PP413-35-G	1.68	32	33.8	66.2	15.2	Abchar 413 resin with "E" glass cloth, Style 181, A1100 finish as reinforcement
382	N151-35-R	–	–	34.2	65.8	–	Imidite 2803 (AFR-151) resin with Refrasil cloth C100-48 as reinforcement
394	N151-35-C	–	–	37.4	62.6	29.7	Imidite 2803 (AFR-151) resin with Carbon cloth CCA-1 as reinforcement
434	DDPO-35-GU	–	–	37.0	63.0	–	Diphenyl oxide resin with Graphite cloth G1550, uncoated, as reinforcement
435	SG7-35-GU	–	–	37.4	62.6	–	Skybond 700 resin with Graphite cloth G1550, uncoated, as reinforcement
436	CP-35-GU	–	–	36.2	63.8	–	Chrome -P resin with Graphite cloth G1550, uncoated, as reinforcement
438	DNB-35-GU	–	–	38.0	62.0	–	2,7 dihydroxynaphthalene phenol formaldehyde resin with Graphite cloth G1550, uncoated, as reinforcement
487	TP(H)-35-GU	–	–	32.6	67.4	–	High Tungsten-P resin with Graphite cloth G1550, uncoated, as reinforcement
488	PPO-35-GU	–	–	35.4	64.6	26.1	Polyphenylene oxide resin with Graphite cloth G1550, uncoated, as reinforcement
506	9-PP700-30-C	–	–	33.0	52.6 Reinforcement 14.4 Filler	27.6	91LD resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
507	PPP-PP700-30-C	–	–	30.9	54.3 Reinforcement 14.8 Filler	31.5	p-Phenylphenol phenol formaldehyde resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
508	D-PP700-30-C	–	–	29.1[1]	55.7 Reinforcement[1] 15.2 Filler[1]	–	DEN438 resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
528	PP412-40-AQ570	1.50	30	42.1	57.9	17.6	Abchar 412 resin with Astroquartz No. 570 as reinforcement

1 The final resin, reinforcement and filler contents could not be determined because of a weight loss due to possible side reactions. Such reactions could take place between the epoxy resin and Abchar 700 or the epoxy resin catalyst and Abchar 700 or both. The values reported are for the laminate as molded.

*For further identification of materials, see Table 14 on page 66.

TABLE 3

PROPERTIES OF LAMINATED SQUARES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
379/394	N151-35-C	1.34	70	37.4	62.6	29.7	Imidite 2803 (AFR-151) resin with Carbon cloth CCA-1 as reinforcement
381/382	N151-35-R	1.40	50	34.2	65.8	34.9	Imidite 2803 (AFR-151) resin with Refrasil cloth C100-48 as reinforcement
443/434	DDPO-35-GU	1.30	43	37.0	63.0	–	Diphenyl oxide resin with Graphite cloth G1550, uncoated, as reinforcement
446/435	SG7-35-GU	1.18	10	37.4	62.6	–	Skybond 700 resin with Graphite cloth G1550, uncoated, as reinforcement
449/436	CP-35-GU	1.34	45	36.2	63.8	–	Chrome-P resin with Graphite cloth G1550, uncoated, as reinforcement
455/438	DNB-35-GU	1.40	45	38.0	62.0	27.7	2,7 dihydroxynaphthalene phenol formaldehyde resin (high barium content catalyst) with Graphite cloth G1550, uncoated, as reinforcement
495/487	TP(H)-35-GU	1.45	82	32.6	67.4	–	High Tungsten-P resin with Graphite cloth G1550, uncoated, as reinforcement
498/488	PPO-35-GU	1.29	30	35.4	64.6	26.1	Polyphenylene oxide resin with Graphite cloth G1550, uncoated, as reinforcement

*For further identification of materials, see Table 14 on page 66.

TABLE 4

PROPERTIES OF PELLET SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
361	PP413-35-C	1.33	58	34.4	65.6	26.3	Abchar 413 resin with Carbon cloth CCA-1 as reinforcement
362	PP413-35-R	1.58	37	37.3	62.7	5.1	Abchar 413 resin with Refrasil cloth C100-48 as reinforcement
377/394	N151-35-C	1.34	70	37.4	62.6	29.7	Imidite 2803 (AFR-151) resin with Carbon cloth CCA-1 as reinforcement
380/382	N151-35-R	1.40	50	34.2	65.8	34.9	Imidite 2803 (AFR-151) resin with Refrasil cloth C100-48 as reinforcement
391	9-35-14	1.17	–	30.6[1]	69.4	53.2[1]	91LD resin with Pyrolytic Graphite No. 14 fibers
397b	GR1-40-C	1.27	40	41.3	58.7	–	GR-1 resin with Carbon cloth CCA-1 as reinforcement
426	PH9-35-R	1.81	85	35.7	64.3	–	PH990 resin with Refrasil cloth C100-48 as reinforcement
427	PH9-35-C	1.47	79	35.8	64.2	–	PH990 resin with Carbon cloth CCA-1 as reinforcement
428	PP500-35-C	1.18	–	33.0	67.0	–	Abchar 500 (high molecular weight poly-phenylene) resin with Carbon cloth CCA-1 as reinforcement
429	PP500-35-R	1.63	–	35.0	65.0	–	Abchar 500 (high molecular weight poly-phenylene) resin with Refrasil cloth C100-48 as reinforcement
431	PP413-PP600-30-C	1.28	30	32.0[2]	53.4 Reinforcement[2] 14.6 Filler[2]	– –	Abchar 413 resin with Carbon cloth CCA-1 as reinforcement and Abchar 600 (para-polyphenylene) as filler
444/434	DDPO-35-GU	1.29	43	37.0	63.0	–	Diphenyl oxide resin with Graphite cloth G1550, uncoated, as reinforcement
447/435	SG7-35-GU	1.19	10	37.4	62.6	–	Skybond 700 resin with Graphite cloth G1550, uncoated, as reinforcement
450/436	CP-35-GU	1.34	45	36.2	63.8	–	Chrome-P resin with Graphite cloth G1550 uncoated, as reinforcement
456/438	DNB-35-GU	1.40	45	38.0	62.0	27.7	2,7 dihydroxynaphthalene phenol formaldehyde resin (high barium content catalyst) with Graphite cloth G1550, uncoated, as reinforcement

*For further identification of materials, see Table 14 on page 66.

TABLE 4 (CONTINUED)

PROPERTIES OF PELLET SPECIMENS

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
460a-1 460a-2	9-28-BF	1.63 1.75	0 0	31.2 30.3	68.8 69.7	– –	91LD resin with boron fibers as reinforcement
460b-1 460b-2	9-28-BF	1.83 1.80	0 0	29.6 29.4	70.4 70.6	– –	91LD resin with boron fibers as reinforcement
465-1 465-2 465-3	D-35-BNF	1.41 1.49 1.46	60 67 64	34.9 34.1 35.0	65.1 65.9 65.0	– – –	91LD resin with boron nitride fibers as reinforcement
477	D-35-R	1.71	–	34.4	65.6	0	DEN 438 resin with Refrasil cloth C100-48 as reinforcement
496/487	TP(H)-35-GU	1.46	82	32.6	67.4	–	High Tungsten-P resin with Graphite cloth G1550, uncoated, as reinforcement
499/488	PPO-35-GU	1.32	30	35.4	64.6	23.2	Polyphenylene oxide resin with Graphite cloth G1550, uncoated, as reinforcement
512/506	9-PP700-30-C	1.32	–	33.0	52.6 Reinforcement 14.4 Filler.	27.6	91LD resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
513/507	PPP-PP700-30-C	1.27	–	30.9	54.3 Reinforcement 14.8 Filler	31.5	p-Phenylphenol phenol formaldehyde resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
514/508	D-PP700-30-C	1.20	–	29.1[3]	55.7 Reinforcement[3] 15.2 Filler[3]	–	DEN 438 resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement

1 Pellets were impregnated with DEN 438 resin to fill voids in specimens. Percentage of voids filled in specimens is as follows:

Specimen No. 1	Specimen No. 2	Specimen No. 3
Effectively 100 percent	75.9	92.5

2 These values are for the unpostcured specimens.

3 The final resin, reinforcement and filler contents could not be determined because of a weight loss due to possible side reactions. Such reactions could take place between the epoxy resin and Abchar 700 or the epoxy resin catalyst and Abchar 700 or both. The values reported are for the laminate as molded.

*For further identification of materials, see Table 14 on page 66.

TABLE 5

PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
397a K-54-1	GR1-40-C	1.12	40	41.3	58.7	–	GR-1 resin with Carbon cloth CCA-1 as reinforcement
403b K-61-2	SG7-40-C	1.28	30	40.2	59.8	–	Skybond 700 resin with Carbon cloth CCA-1 as reinforcement
405 K-63-1 K-63-2	D-30-R	 1.60 1.63	 – –	 32.1 31.9	 67.9 68.1	 15.0 12.6	DEN 438 resin with Refrasil cloth C100-48 as reinforcement
432 K-73-1 K-73-2	PP413-PP600-30-C	 1.18 1.15	 – –	 31.6[1] 31.7[1]	 68.4[1] 68.3[1]	 – –	Abchar 413 resin with Carbon cloth CCA-1 as reinforcement and Abchar 600 (para-polyphenylene) as filler
433 K-74-1 K-74-2	PP413-PP600-30-R	 1.49 1.53	 – –	 28.8[1] 28.8[1]	 55.9[1] 55.9[1]	 – –	Abchar 413 resin with Refrasil cloth C100-48 as reinforcement and Abchar 600 (para-polyphenylene) as filler
433b K-74-3	PP413-PP600-30-R	1.42	–	28.5[1]	71.5[1]	–	Abchar 413 resin with Refrasil cloth C100-48 as reinforcement and Abchar 600 (para-polyphenylene) as filler
466a K-75-1	D-28-BF	2.03	83	21.8- 32.0[2]	78.2- 68.0	– –	DEN 438 resin with boron fibers as reinforcement
466b K-75-2	D-28-BF	1.87	–	32.4- 41.2[2]	67.6- 58.8	– –	DEN 438 resin with boron fibers as reinforcement
489 K-81-1 K-81-2 K-81-3 K-81-4	9-35-AQ570	 1.67 1.70 1.62 1.70	 75 75 75 75	 36.0 36.6 36.4 35.0	 64.0 63.4 63.6 65.0	 – – – –	91LD resin with Astroquartz No. 570 as reinforcement
490 K-82-1 K-82-2 K-82-3 K-82-4	9-35-AS41B	 1.60 1.60 1.58 1.54	 80 80 80 80	 36.5 35.0 35.5 35.5	 63.5 65.0 64.5 64.5	 – – – –	91LD resin with Astrosil 11341-B as reinforcement
509 K-85-1 K-85-2	9-PP700-30-C	 1.44 1.45	 – –	 29.3 29.2	55.6 Reinforcement 15.1 Filler 55.6 Reinforcement 15.2 Filler	 15.9 14.3	91LD resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement

*For further identification of materials, see Table 14 on page 66.

TABLE 5 (CONTINUED)

PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Code Number	Density gms/cc	Barcol Hardness	Composition-Weight-Percent			Description of Material*
				Resin	Reinforcement	Volume-Percent Voids in Resin	
510 K-86-1 K-86-2	PPP-PP700-30-C	 1.42 1.43	 – –	 32.4 32.9	53.1 Reinforcement 14.5 Filler 52.7 Reinforcement 14.4 Filler	 10.9 9.2	p-Phenylphenol phenol formaldehyde resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement
511 K-87-1 K-87-2	D-PP700-30-C	 1.38 1.39	 – –	 30.1 30.0	54.9 Reinforcement 15.0 Filler 55.0 Reinforcement 15.0 Filler	 22.4 21.3	DEN 438 resin with Abchar 700 as filler and Carbon cloth CCA-1 as reinforcement

1 These values are for the unpostcured specimens. The weight-percent of filler content as molded was as follows: Data Sheet No. 432; K-73-1 – 15.4 percent; K-73-2 – 15.3 percent; Data Sheet No. 433; K-74-1 – 15.3 percent; K-74-2 – 15.3 percent; Data Sheet No. 433b; K-74-3 – 19.9 percent.

2 Boron fiber diameters range from 3 to 6 mils with densities of 2.88 gm/cc and 2.46 gm/cc respectively. The range of resin contents calculated assume no voids in the part. The resin content for a 4-mil average fiber diameter is 26.5 weight-percent for K-75-1 and 36.5 weight-percent for K-75-2.

*For further identification of materials, see Table 14 on page 66.

TABLE 6

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
361	Pellet	PP413-35-C	4 Mar 65, Appendix B (1a)	3 May 66	2748. 1/903	8 Jun 66
362	Pellet	PP413-35-R	4 Mar 65, Appendix B (1b)	3 May 66	2748. 1/903	8 Jun 66
363	Laminate	PP413-35-G	4 Mar 65, Appendix B (1c)	3 May 66	2748. 1/903	8 Jun 66
377/394	Pellet	N151-35-C	15 Apr 65 (3a)	14 Oct 66	2748. 1/985	4 Nov 66
378/394	Hot Gas Flow	N151-35-C	15 Apr 65 (3a)	14 Oct 66	2748. 1/985	4 Nov 66
379/394	Lam. Square	N151-35-C	15 Apr 65 (3a)	14 Oct 66	2748. 1/985	4 Nov 66
380/382	Pellet	N151-35-R	15 Apr 65 (3b)	4 Mar 66	2748. 1/882	11 Apr 66
381/382	Lam. Square	N151-35-R	15 Apr 65 (3b)	4 Mar 66	2748. 1/882	11 Apr 66
391	Pellet	9-35-14	5 Mar 65, Appendix C (1b)	4 Mar 66	2748. 1/882	11 Apr 66
397a	Nozzle	GR1-40-C	11 Aug 65, Appendix D (1a)	3 May 66	2748. 1/903	8 Jun 66
397b	Pellet	GR1-40-C	11 Aug 65, Appendix D (1a)	3 May 66	2748. 1/903	8 Jun 66
403b	Nozzle	SG7-40-C	11 Aug 65, Appendix D (1g)	1 Apr 66	2748. 1/882	11 Apr 66
405	Nozzle	D-30-R	11 Aug 65, Appendix D (1i)	1 Apr 66	2748. 1/882	11 Apr 66
426	Pellet	PH9-35-R	7 Jul 65, Appendix A (3a)	1 Apr 66	2748. 1/882	11 Apr 66
427	Pellet	PH9-35-C	7 Jul 65, Appendix A (3b)	1 Apr 66	2748. 1/882	11 Apr 66
428	Pellet	PP500-35-C	11 Aug 65, Appendix A (1a)	20 Jun 66	2748. 1/913	21 June 66
429	Pellet	PP500-35-R	11 Aug 65, Appendix A (1b)	20 Jun 66	2748. 1/913	21 Jun 66
431	Pellet	PP413-PP600-30-C	1 Feb 66, Appendix C (1a)	1 Apr 66	2748. 1/882	11 Apr 66
432	Nozzle	PP413-PP600-30-C	1 Feb 66 Appendix B (1a)	2 Jun 66	2748. 1/882	8 Jun 66
433	Nozzle	PP413-PP600-30-R	1 Feb 66, Appendix B (1b)	1 Apr 66	2748. 1/882	11 Apr 66
433b	Nozzle	PP413-PP600-30-R	1 Feb 66, Appendix B (1b)	1 Apr 66	2748. 1/882	11 Apr 66
442/434	Hot Gas Flow	DDPO-35-GU	1 Feb 66, Appendix A (1a)	4 Mar 66	2748. 1/882	11 Apr 66
443/434	Lam. Square	DDPO-35-GU	1 Feb 66, Appendix A (1a)	4 Mar 66	2748. 1/882	11 Apr 66
444/434	Pellet	DDPO-35-GU	1 Feb 66, Appendix A (1a)	4 Mar 66	2748. 1/882	11 Apr 66
445/435	Hot Gas Flow	SG7-35-GU	1 Feb 66, Appendix A (1b)	4 Mar 66	2748. 1/882	11 Apr 66
446/435	Lam. Square	SG7-35-GU	1 Feb 66, Appendix A (1b)	4 Mar 66	2748. 1/882	11 Apr 66
447/435	Pellet	SG7-35-GU	1 Feb 66, Appendix A (1b)	4 Mar 66	2748. 1/882	11 Apr 66
448/436	Hot Gas Flow	CP-35-GU	1 Feb 66, Appendix A (1c)	4 Mar 66	2748. 1/882	11 Apr 66
449/436	Lam. Square	CP-35-GU	1 Feb 66, Appendix A (1c)	4 Mar 66	2748. 1/882	11 Apr 66
450/436	Pellet	CP-35-GU	1 Feb 66, Appendix A (1c)	4 Mar 66	2748. 1/882	11 Apr 66
454/438	Hot Gas Flow	DNB-35-GU	1 Feb 66, Appendix A (1e)	4 Mar 66	2748. 1/882	11 Apr 66
455/438	Lam. Square	DNB-35-GU	1 Feb 66, Appendix A (1e)	4 Mar 66	2748. 1/882	11 Apr 66
456/438	Pellet	DNB-35-GU	1 Feb 66, Appendix A (1e)	4 Mar 66	2748. 1/882	11 Apr 66
460a	Pellet	9-28-BF	1 Feb 66, Appendix C (1b)	3 May 66	2748. 1/903	8 Jun 66
460b	Pellet	9-28-BF	1 Feb 66, Appendix C (1b)	3 May 66	2748. 1/903	8 Jun 66
465	Pellet	D-35-BNF	21 Apr 66, Appendix B (1a)	3 May 66	2748. 1/903	8 Jun 66
466a	Nozzle	D-28-BF	21 Apr 66, Appendix D (1a)	2 Jun 66	2748. 1/903	8 Jun 66
466b	Nozzle	D-28-BF	21 Apr 66, Appendix D (1a)	20 Jun 66	2748. 1/913	21 Jun 66
477	Pellet	D-35R	21 Apr 66, Appendix B (1c)	19 Jan 67	2748. 1/1030	30 Jan 67
489	Nozzle	9-35-AQ570	21 Apr 66, Appendix D (1b)	20 Jun 66	2748. 1/913	21 Jun 66
490	Nozzle	9-35-AS41B	21 Apr 66, Appendix D (1c)	20 Jun 66	2748. 1/913	21 Jun 66

TABLE 6 (CONTINUED)

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
494/487	Hot Gas Flow	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
495/487	Lam. Square	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
496/487	Pellet	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
497/488	Hot Gas Flow	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
498/488	Lam. Square	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
499/488	Pellet	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
500	Hot Gas Flow	9-35-C	19 Oct 66, Appendix C (1a)	28 Oct 66	2748.1/985	4 Nov 66
501	Hot Gas Flow	170-35-C	19 Oct 66, Appendix C (1c)	22 Nov 66	2748.1/1014	12 Dec 66
502	Hot Gas Flow	171-35-C	19 Oct 66, Appendix C (1b)	22 Nov 66	2748.1/1014	12 Dec 66
503	Hot Gas Flow	172-35-C	19 Oct 66, Appendix C (1d)	22 Nov 66	2748.1/1014	12 Dec 66
504	Hot Gas Flow	PP413-35-C	19 Oct 66, Appendix C (1e)	11 Nov 66	2748.1/1014	12 Dec 66
505	Hot Gas Flow	DP4-35-C	19 Oct 66, Appendix C (1f)	7 Dec 66	2748.1/1030	30 Jan 67
509	Nozzle	9-PP700-30-C	19 Oct 66, Appendix B (1a)	4 Jan 67	2748.1/1030	30 Jan 67
510	Nozzle	PPP-PP700-30-C	19 Oct 66, Appendix B (1b)	4 Jan 67	2748.1/1030	30 Jan 67
511	Nozzle	D-PP700-30-C	19 Oct 66, Appendix B (1c)	4 Jan 67	2748.1/1030	30 Jan 67
512/506	Pellet	9-PP700-30-C	19 Oct 66, Appendix B (1a)	7 Dec 66	2748.1/1030	30 Jan 67
513/507	Pellet	PPP-PP700-30-C	19 Oct 66, Appendix B (1b)	7 Dec 66	2748.1/1030	30 Jan 67
514/508	Pellet	D-PP700-30-C	19 Oct 66, Appendix B (1c)	7 Dec 66	2748.1/1030	30 Jan 67
515/506	Hot Gas Flow	9-PP700-30-C	19 Oct 66, Appendix B (1a)	7 Dec 66	2748.1/1030	30 Jan 67
516/507	Hot Gas Flow	PPP-PP700-30-C	10 Oct 66 Appendix B (1b)	7 Dec 66	2748.1/1030	30 Jan 67
517/508	Hot Gas Flow	D-PP700-30-C	19 Oct 66, Appendix B (1c)	7 Dec 66	2748.1/1030	30 Jan 67
528	Laminate	PP412-40-AQ570	18 Nov 66, Appendix B (4)	19 Jan 67	2748.1/1030	30 Jan 67

TABLE 6 (CONTINUED)

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Code	RTD Letter Reference	Date of Shipment	Detailed Letter Report	
					Reference Number	Date
494/487	Hot Gas Flow	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
495/487	Lam. Square	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
496/487	Pellet	TP (H)-35-GU	21 Apr 66, Appendix C (1a)	20 Oct 66	2748.1/985	4 Nov 66
497/488	Hot Gas Flow	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
498/488	Lam. Square	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
499/488	Pellet	PPO-35-GU	21 Apr 66, Appendix C (1b)	27 Oct 66	2748.1/985	4 Nov 66
500	Hot Gas Flow	9-35-C	19 Oct 66, Appendix C (1a)	28 Oct 66	2748.1/985	4 Nov 66
501	Hot Gas Flow	170-35-C	19 Oct 66, Appendix C (1c)	22 Nov 66	2748.1/1014	12 Dec 66
502	Hot Gas Flow	171-35-C	19 Oct 66, Appendix C (1b)	22 Nov 66	2748.1/1014	12 Dec 66
503	Hot Gas Flow	172-35-C	19 Oct 66, Appendix C (1d)	22 Nov 66	2748.1/1014	12 Dec 66
504	Hot Gas Flow	PP413-35-C	19 Oct 66, Appendix C (1e)	11 Nov 66	2748.1/1014	12 Dec 66
505	Hot Gas Flow	DP4-35-C	19 Oct 66, Appendix C (1f)	7 Dec 66	2748.1/1030	30 Jan 67
509	Nozzle	9-PP700-30-C	19 Oct 66, Appendix B (1a)	4 Jan 67	2748.1/1030	30 Jan 67
510	Nozzle	PPP-PP700-30-C	19 Oct 66, Appendix B (1b)	4 Jan 67	2748.1/1030	30 Jan 67
511	Nozzle	D-PP700-30-C	19 Oct 66, Appendix B (1c)	19 Jan 67	2748.1/1030	30 Jan 67
512/506	Pellet	9-PP700-30-C	19 Oct 66, Appendix B (1a)	7 Dec 66	2748.1/1030	30 Jan 67
513/507	Pellet	PPP-PP700-30-C	19 Oct 66, Appendix B (1b)	7 Dec 66	2748.1/1030	30 Jan 67
514/508	Pellet	D-PP700-30-C	19 Oct 66, Appendix B (1c)	7 Dec 66	2748.1/1030	30 Jan 67
515/506	Hot Gas Flow	9-PP700-30-C	19 Oct 66, Appendix B (1a)	7 Dec 66	2748.1/1030	30 Jan 67
516/507	Hot Gas Flow	PPP-PP700-30-C	19 Oct 66, Appendix B (1b)	7 Dec 66	2748.1/1030	30 Jan 67
517/508	Hot Gas Flow	D-PP700-30-C	19 Oct 66, Appendix B (1c)	7 Dec 66	2748.1/1030	30 Jan 67
528	Laminate	PP412-40-AQ570	18 Nov 66, Appendix B (4)	19 Jan 67	2748.1/1030	30 Jan 67

TABLE 7

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin	Data Sheet Number	Type Specimen
Astroquartz No. 570 (High silica content fabric)	91LD	489	Nozzle
	Polyphenylene (Abchar 412)	528	Lam.
Astrosil 11341-B (High silica content fabric)	91LD	490	Nozzle
Boron Fibers	91LD	460a	Pellet
		460b	Pellet
	DEN 438	466a	Nozzle
		466b	Nozzle
Boron Nitride Fibers	DEN 438	465	Pellet
Carbon Cloth CCA-1	91LD	500	HGF
	91LD with Abchar 700 as filler	509	Nozzle
		512/506	Pellet
		515/506	HGF
	DEN 438 with Abchar 700 as filler	511	Nozzle
		514/508	Pellet
		517/508	HGF
	F170	501	HGF
	F171	502	HGF
	F172	503	HGF
	GR-1	397a	Nozzle
		397b	Pellet
	Imidite 2803 (AFR-151)	377/394	Pellet
		378/394	HGF
		379/394	Lam. Sq.
Carbon Cloth CCA-1	PH 990	427	Pellet
	Phenyl Aldehyde (DP-4-31)	505	HGF
	p-Phenylphenol phenol formaldehyde with Abchar 700 as filler	510	Nozzle
		513/507	Pellet
		516/507	HGF
	Polyphenylene (Abchar 413)	361	Pellet
		504	HGF
	Polyphenylene (Abchar 413) with Abchar 600 as filler	431	Pellet
		432	Nozzle
	Polyphenylene (Abchar 500)	428	Pellet
	Skybond 700	403b	Nozzle
"E" Glass Cloth, Style 181, A1100 Finish	Polyphenylene (Abchar 413)	363	Laminate
Graphite Cloth G1550, uncoated	Chrome-P	448/436	HGF
		449/436	Lam. Sq.
		450/436	Pellet
	2.7 dihydroxynaphthalene phenol formaldehyde (high barium content catalyst)	454/438	HGF
		455/438	Lam. Sq.
		456/438	Pellet
	Polyphenylene Oxide	497/488	HGF
		498/488	Lam. Sq.
		499/488	Pellet
	QX-2682.1	442/434	HGF
		443/434	Lam. Sq.
		444/434	Pellet

TABLE 7 (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin	Data Sheet Number	Type Specimen
Graphite Cloth G1550 uncoated	Skybond 700	445/435	HGF
		446/435	Lam. Sq.
		447/435	Pellet
	Tungsten-P (High)	494/487	HGF
		495/487	Lam. Sq.
		496/487	Pellet
Pyrolytic Graphite No. 14 fibers	91LD	391	Pellet
Refrasil Cloth C100-48 (High silica content fabric)	DEN 438	405	Nozzle
		477	Pellet
	Imidite 2803 (AFR-151)	380/382	Pellet
		381/382	Lam. Sq.
	PH 990	426	Pellet
	Polyphenylene (Abchar 413)	362	Pellet
	Polyphenylene (Abchar 413) with Polyphenylene (Abchar 600) as filler	433	Nozzle
		433b	Nozzle
	Polyphenylene (Abchar 500)	429	Pellet

TABLE 8

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
91LD	Phenolic	Astroquartz No. 570	489	Nozzle
		Astrosil 11341-B	490	Nozzle
		Boron Fibers	460a	Pellet
			460b	Pellet
		Carbon Cloth CCA-1	500	HGF
		Pyrolytic Graphite No. 14 Fibers	391	Pellet
91LD + 8% Abchar 700 as filler	Phenolic with a Polyphenylene filler	Carbon Cloth CCA-1	509	Nozzle
			512/506	Pellet
			515/506	HGF
Chrome-P	Chrome based metal organic phenolic	Graphite Cloth G1550, uncoated	448/436	HGF
			449/436	Lam. Sq.
			450/436	Pellet
DEN 438	Epoxy novolac	Boron Fibers	466a	Nozzle
			466b	Nozzle
		Boron Nitride Fibers	465	Pellet
		Refrasil Cloth C100-48	405	Nozzle
			477	Pellet
DEN 438 with Abchar 700 as filler	Epoxy novolac with a Polyphenylene filler	Carbon Cloth CCA-1	511	Nozzle
			514/508	Pellet
			517/508	HGF
27 Dihydroxynaphthalene phenol formaldehyde (high barium content catalyst)	Modified Phenolic	Graphite Cloth G1550, uncoated	454/438	HGF
			455/438	Lam. Sq.
			456/438	Pellet

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
F170	Polyimide	Carbon Cloth CCA-1	501	HGF
F171	Polyarylene	Carbon Cloth CCA-1	502	HGF
F172	Diphenyloxide	Carbon Cloth CCA-1	503	HGF
GR-1	Proprietary composition	Carbon Cloth CCA-1	397a	Nozzle
			397b	Pellet
Imidite 2803 (AFR-151)	Polybenzimidazole (Amide blocked)	Carbon Cloth CCA-1	377/394	Pellet
			378/394	HGF
			379/394	Lam. Sq.
		Refrasil Cloth C100-48	380/382	Pellet
			381/382	Lam. Sq.
PH990	Phosphonitrilic-modified phenolic	Carbon Cloth CCA-1	427	Pellet
		Refrasil Cloth C100-48	426	Pellet
Phenyl Aldehyde (DP-4-31)	Modified phenolic	Carbon Cloth CCA-1	505	HGF
p-Phenylphenol phenol formaldehyde with Abchar 700 as filler	Modified phenolic with a Polyphenylene filler	Carbon Cloth CCA-1	510	Nozzle
			513/507	Pellet
			516/507	HGF
Polyphenylene (Abchar 412)	m-Polyphenylene	Astroquartz No. 570	528	Lam.
Polyphenylene (Abchar 413)	m-Polyphenylene	Carbon Cloth CCA-1	361	Pellet
			504	HGF
		"E" Glass Cloth, Style 181, A1100 Finish	363	Lam.

TABLE 8 (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
Polyphenylene (Abchar 413)	m-Polyphenylene	Refrasil Cloth C100-48	362	Pellet
Polyphenylene (Abchar 413) with polyphenylene (Abchar 600) as filler	m-Polyphenylene with para-polyphenylene as filler	Carbon Cloth CCA-1	431	Pellet
			432	Nozzle
		Refrasil Cloth C100-48	433	Nozzle
			433b	Nozzle
Polyphenylene (Abchar 500)	High molecular weight m-Polyphenylene without catalyst	Carbon Cloth CCA-1	428	Pellet
		Refrasil Cloth C100-48	429	Pellet
Polyphenylene Oxide	Polyphenylene Oxide	Graphite Cloth G1550, uncoated	497/488	HGF
			498/488	Lam. Sq.
			499/488	Pellet
QX-2682.1	Diphenyl Oxide	Graphite Cloth G1550, uncoated	442/434	HGF
			443/434	Lam. Sq.
			444/434	Pellet
Skybond 700	Polyimide	Carbon Cloth CCA-1	403b	Nozzle
		Graphite Cloth G1550, uncoated	445/435	HGF
			446/435	Lam. Sq.
			447/435	Pellet
Tungsten-P (High)	Tungsten based metal organic phenolic	Graphite Cloth G1550, uncoated	494/487	HGF
			495/487	Lam. Sq.
			496/487	Pellet

TABLE 9

FABRICATION DETAILS – HOT GAS FLOW TYPE D[1] SPECIMENS

Data Sheet Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			†Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
378/394	8" x 7" x 1/2"	N151-35-C	Used as received	Preimpregnated by Narmco	–	–	–	700	500	180	H-3
442/434	9" x 6-3/4" x 1/2"	DDPO-35-GU	1/1.3 acetone	Spatula coating	30	60 min at 160°F	5 min at 225°F	350	500	120	B-6
445/435	9" x 6-3/4" x 1/2"	SC7-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 200°F[2]	30 min at 250°F	600	104	180	E
448/436	9" x 6-3/4" x 1/2"	CP-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 180°F	–	180-250	104	120-360	J-1
454/438	9" x 6-3/4" x 1/2"	DNB-35-GU	1/0.5 acetone	Spatula coating	20	20 min at 160°F	20 min at 220°F	300	300	120	B-1
494/487	9" x 6-1/2" x 1/2"	TP(H)-35-GU	1/1 acetone	Brush coating	60	60 min at 160°F	20 min at 225°F	180-250	500	45-120	J-3
497/487	9" x 6-1/2" x 1/2"	PPO-35-GU	1/2.7 xylene	Spatula coating	60	60 min at 160°F 60 min at 250°F	–	600	500	180	None
500	18" x 16-3/4" x 1/2"	9-35C	1/0.5 acetone	Brush coating	60	60 min at 160°F	20 min at 225°F	300	500	180	B-1
501	8-1/2" x 6" x 1/2"	170-35-C	1/0.4 acetone	Brush coating	60	60 min at 200°F	30 min at 250°F[2]	600	300	120	B-7
502	8-1/2" x 6" x 1/2"	171-35-C	1/0.5 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	350	300	120	B-7
503	8-1/2" x 6" x 1/2"	172-35-C	1/0.5 acetone	Brush coating	60	60 min at 160°F	25 min at 225°F	350	300	120	B-8
504	8-1/2" x 6" x 1/2"	PP413-35-C	Used as received	Brush coating	30	20 min at 160°F[2]	–	400	300	120	J-3
505	8" x 6-1/2" x 1/2"	DP4-35-C	1/1 acetone	Spatula coating	60	60 min at 160°F	30 min at 225°F	300	500	120	B-1
515/506	8" x 6-1/2" x 1/2"	9-PP700-30-C	1/1 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	300	120	B-1
516/507	8" x 6-1/2" x 1/2"	PPP-PP700-30-C	1/1 acetone	Spatula coating	60	60 min at 160°F	15 min at 240°F	300	300	120	B-1
517/508	8" x 6-1/2" x 1/2"	D-PP700-30-C	1/0.5 acetone	Spatula coating		30 min at 160°F	15 min at 225°F	300	300	120	B-1

TABLE 9 (CONTINUED)

FABRICATION DETAILS – HOT GAS FLOW TYPE D[1] SPECIMENS

1 Dimensions for Type D hot gas flow specimens are 3.333" ± 0.010" x 2.000" ± 0.010" x 0.502" ± 0.002"

2 Followed by 16 hours vacuum dry at room temperature

† The following postcure schedules were used:

B-1 18 hours at 275°F, 72 hours from 275°F to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.

B-6 At the start of the postcure cycle, parts were placed in the oven at room temperature. The oven temperature was then raised to 275°F over a period of 12 to 16 hours. The remainder of the cycle consisted of 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 9 hours from 400° to 490°F, 2 hours at 500°F, 7 hours cooling to below 200°F.

B-7 24 hours at each of the following temperatures: 375°, 435°, 475° and 575°F, 4 hours at 700°F, 6 hours between temperatures. Cool to below 200°F. Parts were postcured in argon.

B-8 16 hours at 275°F, 72 hours from 275° to 400°F, 6 hours from 400° to 450°F, 4 hours at 450°F, 6 hours from 450° to 500°F, 12 hours at 500°F. Cool to below 200°F.

E 24 hours at 375°F, 24 hours at 435°F, 24 hours at 475°F, 24 hours at 575°F (6 hours between temperatures), 7 hours cooling to below 200°F.

H-3 24 hours room temperature to 600°F, 24 hours at 600°F, 10 hours from 600° to 650°F, 24 hours at 650°F, 10 hours from 650° to 700°F, 24 hours at 700°F, 10 hours from 700° to 750°F, 6 hours at 750°F, 10 hours from 750° to 800°F, 6 hours at 800°F, 10 hours from 800° to 850°F, 6 hours at 850°F, 10 hours cooling to below 200°F. Parts were postcured in argon atmosphere.

I-3 18 hours at 275°F, 108 hours from 275° to 550°F, 6 hours at 550°F, 7 hours cooling to below 200°F. Parts were postcured in argon.

J-1 1 hour at 150°F, 1 hour at 200°F, 1 hour at 250°F, 1 hour at 300°F, 1 hour at 350°F, 2 hours at 400°F, 7 hours cooling to below 200°F.

J-3 24 hours from 100° to 300°F, 1 hour at 300°F, 3 hours from 300° to 350°F, 1 hour at 350°F, 3 hours from 350° to 400°F, 2 hours at 400°F, 7 hours cooling to below 200°F.

TABLE 10

FABRICATION DETAILS – LAMINATES

Data Sheet Number	Dimensions of Laminate	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			† Postcure
					Air Dry Min.	Oven Dry		Temp °F	Pressure PSI	Time Min	
363	6" x 6" x 1/8"	PP413-35-G	Used as received	Dip coating	20	20 min at 160°F	–	400	300	120	I-3
382	7-1/2" x 5" x 1/2"	N151-35-R	Not applicable	Preimpregnated by Narmco	–	–	–	700	200	180	H-1
394	8" x 7" x 1/2"	N151-35-C	Not applicable	Preimpregnated by Narmco	–	–	–	700	500	180	H-3
434	9" x 6-3/4" x 1/2"	DDPO-35-GU	1/1.3 acetone	Spatula coating	30	60 min at 160°F[1]	5 min at 255°F	350	200	120	B-6
435	9" x 6-3/4" x 1/2"	SG7-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 200°F	30 min 250°F	600	104	180	E
436	9" x 6-3/4" x 1/2"	CP-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 180°F	–	180-250	104	120-300	J-1
438	9" x 6-3/4" x 1/2"	DNB-35-GU	1/0.5 acetone	Spatula coating	20	20 min at 160°F	20 min at 220°F	300	300	120	B-1
487	9" x 6-1/2" x 1/2"	TP(H)-35-GU	1/1 acetone	Brush coating	60	60 min at 160°F	20 min at 225°F	180-250	500	45-120	J-3
488	9" x 6-1/2" x 1/2"	PPO-35-GU	1/2.7 xylene	Spatula coating	60	60 min at 160°F	–	600	500	180	None
						60 min at 250°F					
506	8" x 6-1/2" x 1/2"	9-PP700-30-C	1/1 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	300	120	B-1
507	8" x 6-1/2" x 1/2"	PPP-PP700-30-C	1/1 acetone	Spatula coating	60	20 min at 160°F	15 min at 240°F	300	300	120	B-1
508	8" x 6-1/2" x 1/2"	D-PP700-30-C	1/0.5 acetone	Spatula coating	60	30 min at 160°F	20 min at 225°F	300	300	120	B-3
528	7" x 7" x 1/8"	PP412-40-AO570	Used as received	Dip coating	30	25 min at 160°F[1]	–	400	300	120	I-3

TABLE 10 (CONTINUED)

FABRICATION DETAILS – LAMINATES

[1] Followed by 16-18 hours vacuum dry at room temperature.

† The following postcure schedules were used:

B-1	18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.
B-3	17 hours at 275°F, 6 hours from 275°F to 400°F, 1 hour at 400°F, 7 hours cooling to below 200°F.
B-6	At the start of the postcure cycle, parts were placed in the oven at room temperature. The oven temperature was then raised to 275°F over a period of 16 hours. The remainder of the cycle consisted of 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 9 hours 410° to 490°F, 2 hours at 500°F, 7 hours cooling to below 200°F.
E	24 hours at 375°F, 24 hours at 435°F, 24 hours at 475°F, 24 hours at 575°F (6 hours between temperatures), 7 hours cooling to below 200°F.
H-1	24 hours at 600°F, 24 hours at 650°F, 24 hours at 700°F, 6 hours at 750°F. 6 hours at 800°F, 6 hours at 850°F (1-1/2 hours between temperatures), 18 hours cooling to room temperature. Parts were postcured in argon.
H-3	24 hours room temperature to 600°F, 24 hours at 600°F, 10 hours from 600° to 650°F, 24 hours at 650°F, 10 hours from 650° to 700°F, 24 hours at 700°F, 10 hours from 700° to 750°F, 6 hours at 750°F, 10 hours from 750° to 800°F, 6 hours at 800°F, 10 hours from 800° to 850°F, 6 hours at 850°F, 10 hours cooling to below 200°F. Parts were postcured in argon atmosphere
I-3	18 hours at 275°F, 108 hours from 275° to 550°F, 6 hours at 550°F, 7 hours cooling to below 200°F. Specimens were postcured in argon.
J-1	1 hour at 150°F, 1 hour at 200°F, 1 hour at 250°F, 1 hour at 300°F, 1 hour at 350°F, 2 hours at 400°F, 7 hours cooling to below 200°F.
J-3	24 hours from 100° to 300°F, 1 hour at 300°F, 3 hours from 300° to 350°F, 1 hour at 350°F, 3 hours from 350° to 400°F, 2 hours at 400°F, 7 hours cooling to below 200°F.

TABLE 11

FABRICATION DETAILS – LAMINATED SQUARES[1]

Data Sheet Number	Dimensions of Original Laminate	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			Postcure
					Air Dry Min	Oven Dry		Temp °F	Pressure PSI	Time Min	
379/394	8" x 7" x 1/2"	N151-35-C	Used as received	Preimpregnated by Narmco	–	–	–	700	500	180	H-3
381/382	7-1/2" x 5" x 1/2"	N151-35-R	Used as received	Preimpregnated by Narmco	–	–	–	700	200	180	H-1
443/434	9" x 6-3/4" x 1/2"	DDPO-35-GU	1/1.3 acetone	Spatula coating	30	60 min at 160°F	5 min at 225°F	350	200	120	B-6
446/435	9" x 6-3/4" x 1/2"	SG7-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 200°F[2]	30 min at 250°F	600	104	180	E
449/436	9" x 6-3/4" x 1/2"	CP-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 180°F	–	180-250	104	120-300	J-1
455/438	9" x 6-3/4" x 1/2"	DNB-35-GU	1/0.5 acetone	Spatula coating	20	20 min at 160°F	20 min at 220°F	300	300	120	B-1
495/487	9" x 6-1/2" x 1/2"	TP(H)-35-GU	1/1 acetone	Brush coating	60	60 min at 160°F	20 min at 225°F	180-250	500	45-120	J-3
498/488	9" x 6-1/2" x 1/2"	PPO-35-GU	1/2.7 xylene	Spatula coating	60	60 min at 160°F 60 min at 250°F	–	600	500	180	None

[1] Dimensions for laminated squares are 2.000" ± 0.010" x 2.000" ± 0.010" x 0.502" ± 0.002".

[2] Followed by a 16 hour vacuum dry at room temperature

The following postcure schedules were used:

B-1 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.

B-6 At the start of the postcure cycle, parts were placed in the oven at room temperature. The oven temperature was then raised to 275°F over a period of 12 to 16 hours. The remainder of the cycle consisted of 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 9 hours 400° to 490°F, 2 hours at 500°F, 7 hours cooling to below 200°F.

E 24 hours at 375°F, 24 hours at 435°F, 24 hours at 475°F, 24 hours at 575°F (6 hours between temperatures), 7 hours cooling to below 200°F.

H-1 24 hours at 600°F, 24 hours at 650°F, 24 hours at 700°F, 6 hours at 750°F, 6 hours at 800°F, 6 hours at 850°F (1-1/2 hours between temperatures), 18 hours cooling to room temperature. Parts postcured in argon.

H-3 24 hours room temperature to 600°F, 24 hours at 600°F, 10 hours from 600° to 650°F, 24 hours at 650°F, 10 hours from 650° to 700°F, 24 hours at 700°F, 10 hours from 700° to 750°F, 6 hours at 750°F, 10 hours from 750° to 800°F, 6 hours at 800°F, 10 hours from 800° to 850°F, 6 hours at 850°F, 10 hours cooling to below 200°F. Parts were postcured in argon atmosphere.

J-1 1 hour at 150°F, 1 hour at 200°F, 1 hour at 250°F, 1 hour at 300°F, 1 hour at 350°F, 2 hours at 400°F, 7 hours cooling to below 200°F.

J-3 24 hours from 100° to 300°F, 1 hour at 300°F, 3 hours from 300° to 350°F, 1 hour at 350°F, 3 hours from 350° to 400°F, 2 hours at 400°F, 7 hours cooling to below 200°F.

TABLE 12

FABRICATION DETAILS—PELLET SPECIMENS[1]

Data Sheet Number	Dimensions of Original Molding or Laminate	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			† Postcure
					Air Dry Min	Oven Dry		Temp °F	Pressure PSI	Time Min	
361	3-1/2" dia x 1/2"	PP413-35-C	Used as received	Dip coating	20	20 min at 160°F	–	400	1000	120	I-3
362	2" dia x 1/2"	PP413-35-R	Used as received	Dip coating	20	20 min at 160°F	–	400	3200	120	I-3
377/394	8" x 7" x 1/2"	N151-35-C	Used as received	Preimpregnated by Narmco	–	–	–	700	500	180	H-3
380, 382	7-1/2" x 5" x 1/2"	N151-35-R	Used as received	Preimpregnated by Narmco	–	–	–	700	200	180	H-1
391	2" dia x 1/2"	9-35-14	1/7 acetone	Soaking	60[2]	60 min at 160°F	30 min at 225°F	300	200	120	B-1
397b	1.675" dia disc[3]	GR1-40-C	1/2.6 ethyl alcohol	Spatula coating	120[4]	–	360 min at 200°F under vacuum	400-500	1300	120-300	K-1
426	2" dia x 1/2"	PH9-35-R	1/2 acetone	Spatula coating	30	60 min at 160°F	10 min at 200°F	300-450[5]	6400	60	B-2[6]
427	2" dia x 1/2"	PH9-35-C	1/2 acetone	Spatula coating	30	60 min at 160°F	10 min at 200°F	300-450[5]	6400	60	B-2[6]
428	4" x 3" x 1/2"	PP500-35-C	Used as received	Dry powder layup	–	–	–	600	300	120	None
429	3" x 2" x 1/2"	PP500-35-R	Used as received	Dry power layup	–	–	–	600	300	120	None
431	3-1/2" dia x 1/2"	PP413-PP600-30-C	No solvent added	Dip coating	30	10 min at at 160°F	–	400	3500	120	I-3
444/434	9" x 6-3/4" x 1/2"	DDPO-35-GU	1/1.3 acetone	Spatula coating	30	60 min at 160°F	5 min at 225°F	350	200	120	B-6[6]
447/435	9" x 6-3/4" x 1/2"	SG7-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 200°F[4]	30 min at 250°F	600	104	180	E
450/436	9" x 6-3/4" x 1/2"	CP-35-GU	1/0.5 acetone	Spatula coating	960	60 min at 180°F	–	180-250	104	120-300	J-1
456/438	9" x 6-3/4" x 1/2"	DNB-35-GU	1/0.5 acetone	Spatula coating	20	20 min at 160°F	20 min at 220°F	300	300	120	B-1
460a-1	1" x 1" x 1/2"	9-28-BF	Used as received	91LD-boron fiber prepreg received from U.S. Polymeric	–	–	300 min at 200°F in mold	300	500	120	B-1
460a-2							60 min at 200°F in mold	300	500	120	B-1
460b-1	1" x 1" x 3/4"	9-28BF	Used as received	91LD-boron fiber prepreg received from U.S. Polymeric	–	–	60 min at 200°F in mold	300	500	120	B-1
460b-2							30 min at 200°F in mold	300	500	120	B-1

TABLE 12 (CONTINUED)

FABRICATION DETAILS – PELLET SPECIMENS [1]

Data Sheet Number	Dimensions of Original Molding or Laminate	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			† Postcure
					Air Dry Min	Oven Dry		Temp °F	Pressure PSI	Time Min	
465-1 465-2 465-3	3/4" dia x 1/2"	D-35-BNF	–	Soaking and Buchner funnel	–	30 min at 160°F	30 min at 225°F	300	3300	160 120 120	B-1
477	2" dia x 1/2" disc	D-35-R	1/0.4 acetone	Spatula coating	60	30 min at 160°F	30 min at 225°F	300	3300	120	B-3
496/487	9" x 6-1/2" x 1/2"	TP(H)-35-GU	1/1 acetone	Brush coating	60	60 min at 160°F	20 min at 225°F	180-250	500	45-120	J-3
499/488	9" x 6-1/2" x 1/2"	PPO-35-GU	1/2.7 xylene	Spatula coating	60	60 min at 160°F 60 min at 250°F	–	600	500	180	None
512/506	8" x 6-1/2" x 1/2"	9-PP700-30-C	1/1 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	300	120	B-1
513/507	8" x 6-1/2" x 1/2"	PPP-PP-700-30-C	1/1 acetone	Spatula coating	60	20 min at 160°F	15 min at 240°F	300	300	120	B-1
514/508	8" x 6-1/2" x 1/2"	D-PP700-30-C	1/0.5 acetone	Spatula coating	60	30 min at 160°F	20 min at 225°F	300	300	120	B-3

[1] Dimensions for pellet specimens are 0.750" ± 0.010" diameter x 0.502" ± 0.002".

[2] Prepreg was vacuum dried 7 hours at room temperature before oven drying.

[3] Specimens were machined from a broken rocket nozzle insert.

[4] Prepreg was dried at room temperature and under vacuum for an additional 16 hours.

[5] Plies were placed in a 300°F mold. The mold temperature was raised to 450°F over a 6 minute period using a muffler heater.

[6] At the start of postcure cycle, parts were placed in the oven at room temperature. The oven temperature was then raised to 275°F over period of 12 to 16 hours.

† The following postcure schedules were used:

B-1 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.

B-2 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 4 hours at 425°F, 7 hours cooling to below 200°F.

B-3 17 hours at 275°F, 6 hours from 275° to 400°F, 1 hour at 400°F, 7 hours cooling to below 200°F.

B-6 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 9 hours 400° to 490°F, 2 hours at 500°F, 7 hours cooling to below 200°F.

E 24 hours at 375°F, 24 hours at 435°F, 24 hours at 475°F, 24 hours at 575°F (6 hours between temperatures), 7 hours cooling to below 200°F.

TABLE 12 (CONTINUED)

FABRICATION DETAILS – PELLET SPECIMENS[1]

H-1	24 hours at 600°F, 24 hours at 650°F, 24 hours at 700°F, 6 hours at 750°F, 6 hours at 800°F, 6 hours at 850°F (1-1/2 hours between temperatures) 18 hours cooling to below 200°F). Parts were postcured in argon.
H-3	24 hours room temperature to 600°F, 24 hours at 600°F, 10 hours from 600° to 650°F, 24 hours at 650°F, 10 hours from 650° to 700°F, 24 hours at 700°F, 10 hours from 700° to 750°F, 6 hours at 750°F, 10 hours from 750 to 800°F, 6 hours at 800°F, 10 hours from 800° to 850°F, 6 hours at 850°F, 10 hours cooling to below 200°F. Parts were postcured in argon atmosphere.
I-3	18 hours at 275°F, 108 hours from 275°F to 550°F, 6 hours at 550°F, 7 hours cooling to below 200°F. Specimens were postcured in argon.
J-1	1 hour at 150°F, 1 hour at 200°F, 1 hour at 250°F, 1 hour at 300°F, 1 hour at 350°F, 2 hours at 400°F, 7 hours cooling to below 200°F.
J-3	24 hours from 100° to 300°F, 1 hour at 300°F, 3 hours from 300° to to 350°F, 1 hour at 350°F, 3 hours from 350° to 400°F, 2 hours at 400°F, 7 hours cooling to below 200°F.
K-1	18 hours room temperature to 275°F, 144 hours 275° to 500°F, 7 hours cooling to below 200°F. Parts were postcured in argon atmosphere.

TABLE 13

FABRICATION DETAILS – ROCKET NOZZLES[1]

Data Sheet Number	Nozzle Number	Dimensions of Original Molding or Laminate	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		"B" Staging Conditions	Molding Conditions			†Postcure
						Air Dry Min	Oven Dry		Temp °F	Pressure PSI	Time Min	
397a	K-54-1	5" x 3" x 2-1/4"	GR1-40-C	1/2.6 ethyl alcohol	Spatula coating	120[2]	–	360 min at 200°F under vacuum	400-500	1,300	120-300	K-1
403b	K-61-2	5" x 3" x 2-1/4"	SG7-40-C	1/0.5 acetone	Spatula coating	60[2]	60 min at 200°F	30 min at 250°F	600	5,000	240	E
405	K-63-1 K-63-2	1-11/16" dia x 2-1/4"	D-30-R	–	Spatula coating	30	60 min at 160°F	30 min at 225°F	300	4,823	120	B-3
432	K-73-1 K-73-2	1-11/16" dia x 2-1/4"	PP413-PP600-30-C	No solvent added[3]	Dip coating	30	10 min at 160°F	–	400	10,000	120	I-3
433	K-74-1 K-74-2	1-11/16" dia x 21/4"	PP413-PP600-30-R	No solvent added[3]	Dip coating	20	10 min at 160°F	–	400	3,500	120	I-3
433b	K-74-3	1-11/16" dia x 2-1/4"	PP413-PP600-30-R	No solvent added[3]	Dip coating	30	10 min at 160°F	–	400	10,000	120	I-3
466a	K-75-1	1-3/4" x 1-3/4" x 3"	D-28-BF	Not applicable	DEN 438-boron fiber prepreg received AFML	–	–	–	300	500	120	B-3
466b	K-75-2	1-3/4" x 1-3/4" x 2-3/4"	D-28-BF	Not applicable	DEN438-boron fiber prepreg received AFML	– 60	– 60 min at 160 F	– 30 min at 225 F	300 300	500 10,000	120 120	B-3 B-1
489	K-81-1 K-81-2 K-81-3 K-81-4	1-11/16" dia x 2-1/4"	9-35-AQ570	1/0.5 acetone	Spatula coating							
490	K-82-1 K-82-2 K-82-3 K-82-4	1-11/16" dia x 2-1/4"	9-35-AS41B	1/0.5 acetone	Spatula coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
509	K-85-1 K-85-2	1-11/16" dia x 2-1/4"	9-PP700-30-C	1/1 acetone[3]	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	10,000	120	B-1
510	K-86-1 K-86-2	1-11/16" dia x 2-1/4"	PPP-PP700-30-C	1/1.5 acetone[3]	Spatula coating	60	20 min at 160°F	15 min at 240°F	300	10,000	120	B-1
511	K-87-1 K-87-2	1-11/16" dia x 2-1/4"	D-PP700-30-C	1/1 acetone[3]	Spatula coating	60	20 min at 160°F	20 min at 225°F	300 350 400	10,000	120 16(hrs) 60	Combined cure and postcure cycle in press under pressure

TABLE 13 (CONTINUED)

FABRICATION DETAILS – ROCKET NOZZLES[1]

[1] All rocket nozzle inserts were machined to the ASD No. 4 configuration.
[2] Followed by a 16 hour vacuum dry at room temperature.
[3] Resin and filler were blended 15 minutes in a Waring Blendor.
† The following postcure schedules were used:

B-1 18 hours at 275°F, 72 hours from 275° to 400°F, 4 hours at 400°F, 7 hours cooling to below 200°F.

B-3 17 hours at 275°F, 6 hours from 275° to 400°F, 1 hour at 400°F, 7 hours cooling to below 200°F.

E 24 hours at 375°F, 24 hours at 435°F, 24 hours at 475°F, 24 hours at 575°F, (6 hours between temperatures), 7 hours cooling to below 200°F.

I-3 18 hours at 275°F, 108 hours from 275° to 550°F, 6 hours at 550°F. Cool to below 200°F. Specimens were postcured in argon.

K-1 18 hours room temperature to 275°F, 144 hours 275° to 500°F, 7 hours cooling to below 200°F. Specimens were postcured in argon.

TABLE 14

MATERIAL SOURCES

Trade Name or Designation	Type of Material	Source
91LD	Resin	American Reinforced Sales
Astroquartz No. 570	Reinforcement	AFML (J. P. Stevens)
Astrosil 11341-B	Reinforcement	AFML (J. P. Stevens)
Boron fibers (impregnated with 91LD resin)	Prepreg	U.S. Polymeric Chemicals
Boron fibers (impregnated with DEN 438 resin)	Prepreg	AFML
Boron nitride fibers	Reinforcement	AFML
Carbon cloth CCA-1	Reinforcement	HITCO
Chrome-P (Chrome Phenolic)	Resin	Thermo Resist
DEN 438 (Epoxy Novolac)	Resin	Dow Chemical
2, 7 dihydroxynaphthalene phenol formaldehyde (high barium content catalyst)	Resin	Hughes Aircraft
"E" Glass Cloth, Style 181, A1100 finish	Reinforcement	Hastings Plastics
F170 (Imide)	Resin	Coast Manufacturing & Supply
F171 (Polyarylene Phenolic)	Resin	Coast Manufacturing & Supply
F172 (Polyphenylene Phenolic)	Resin	Coast Manufacturing & Supply
GR-1(Organo Phosphonitrilic)	Resin	W. R. Grace
Graphite cloth G1550, uncoated	Reinforcement	HITCO
Imidite 2803 (AFR-151)	Resin	AFML (Narmco)
PH990 (Organo Phosphonitrilic)	Resin	El Monte Chemical
Phenyl aldehyde (DP-4-31)	Resin	Ironsides Resins
p-Phenylphenol phenol formaldehyde	Resin	Hughes Aircraft
Polyphenylene		
Abchar 412*	Resin	
Abchar 413*	Resin	
Abchar 500*	Resin	Hughes Aircraft
Abchar 600*	Resinous filler	
Abchar 700*	Resinous filler	
Polyphenylene Oxide	Resin	General Electric
Pyrolytic Graphite No. 14 fibers	Reinforcement	AFML
QX-2682. 1 (Diphenyl Oxide)	Resin	Dow Chemical
Refrasil cloth C100-48	Reinforcement	HITCO
Skybond 700 (Polyimide)	Resin	Monsanto
Tungsten P (High) (Tungsten Phenolic)	Resin	Thermo Resist

*Information on these resins are given on pages 39, 40 and 41.

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11. Supplementary Notes	12. Sponsoring Military Activity Nonmetallic Materials Division Air Force Materials Laboratory Wright-Patterson AFB, Ohio 45433

13. Abstract

Precise processing techniques were used in preparing new ablative plastic composites. This research involved the use of novel heat resistant resins such as: cross-linked, branched polyphenylenes; para-polyphenylenes, and amide-blocked polybenzimidazole, a phosphonitrilic-modified phenolic, a polyimide, a chrome based metal organic phenolic, a tungsten based metal organic phenolic and a polyphenylene oxide.

Novel materials used as reinforcements included boron fibers, boron nitride fibers, and pyrolytic graphite fibers.

Resin impregnation techniques used in preparing research specimens included spatula or brush coating, dip coating, Buchner funnel impregnation and dry powder layup.

Research specimens were prepared in various geometric configurations depending on their intended ablative characterization. Test specimen geometries included small pellets (3/4-inch diameter by 1/2-inch thick) for hyperthermal reentry screening, subscale rocket nozzles for solid- and liquid-propellant rocket motor screening and hot-gas flow specimens for channel-type hyperthermal tests.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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